THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

MARCH, 1958

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Metal Powders: Symposium

An Informal Discussion on 'Developments in the Production and Quality of Metal Powders' was held on Dec. 4th, 1957, under the auspices of the Powder Metallurgy Joint Group of the Iron and Steel Institute and the Institute of Metals.

Papers presented for discussion dealt with production of powders of some of the reactive metals, electrolytic production of straight and alloyed metal powders, mechanical methods as used in the carbide industry, electrolytic copper powder, atomization of metal and alloy powders, and powders produced by gaseous reduction of aqueous solutions.

The papers, together with discussion, will be published in due course, and will be abstracted in *The Nickel Bulletin* at the time of publication.

Surface Analysis of Metal Powders

See abstract on p. 69.

Vacuum Melting of Alloys

See abstract on p. 81.

Nomenclature of Alloy Phases

See abstract on p. 75.

NICKEL

Sintering of Nickel Oxide at Nicaro

J. A. POLL: 'Sintering of Nickel Oxide at Nicaro, Cuba.'

Paper presented to First Annual Meeting of Society of Mining Engineers of the American Institute of Mining, Metallurgical and Petroleum Engineers, Oct. 15, 1957; 6 pp.

The extraction of nickel from laterite and serpentine nickeliferous ores at the Nicaro plant of the Nickel Processing Corporation involves many complex processes. The low-grade ore, mined by open-pit methods, is dried, ground, roasted under reducing conditions and subjected to ammoniacal leaching, which dissolves the extractable nickel. The dissolved nickel is then precipitated as a basic nickel carbonate, which is calcined at a high temperature to produce a nickel oxide. When the plant first came into production the nickel was acceptable in this uneconomical form but only by virtue of the urgent wartime need for the metal. In 1953, investigations by the National Lead Company, under commission from the General Services Administration, confirmed the feasibility of adapting conventional sintering processes to the production of a nickel-oxide sinter product from the Nicaro ore. The paper describes, with the aid of a flow sheet, the procedure used in the sintering plant which was put into operation in 1954. The raw materials for the process consist of nickel oxide, anthracite coal, return fines and dust, and sintering has the dual effect of producing the nickel oxide in a form capable of easier utilization by industry, whilst also increasing the nickel content of the oxide from 77 to 90 per cent.

Extraction of Nickel and Cobalt: Bibliography

R. B. BAUDER: 'Bibliography on Extractive Metallurgy of Nickel and Cobalt, January 1929–July 1955.' *U.S. Bur. Mines, Information Circular* 7805, Nov. 1957; 159 pp.

The volume contains more than 1600 *annotated abstracts* covering the period specified: it was prepared as part of the U.S. Bureau of Mines research programme on Cuban lateritic and serpentine ores. The preface outlines the scope of the literature search which was made in compilation of the bibliography.

Arrangement of the material is in five sections, covering, respectively, general metallurgy, pyrometallurgical processes, hydrometallurgical processes, electrometallurgical processes, and miscellaneous relevant information. Each section is suitably subdivided. It is noted that the review does not contain references to mining, mineralogy or geology as such, nor statements on production, reserves, etc.

A supplement now in preparation will cover the period 1900–1929.

Vapour Pressure of Liquid Nickel

J. P. MORRIS, G. R. ZELLARS, S. L. PAYNE and R. L. KIPP: 'Vapour Pressures of Liquid Iron and Liquid Nickel.' *U.S. Bur. Mines, Report of Investigations* 5364, Sept. 1957; 10 pp.

A recent investigation by two of the present authors resulted in development of a gas-transport method for measurement of vapour pressures of liquid metals, and the technique was successfully applied to determination of the vapour pressures of copper. The same procedure has now been used for measurements on liquid iron and nickel: the study is part of a project for determining thermodynamic activities in alloys of iron at steelmaking temperatures.

The vapour pressures of iron and nickel were determined at temperatures between 1540° and

1620°C. The results can be represented by the following equations:

$$\log p_{mm}\ (\text{iron}) = -19{,}460/T + 9{\cdot}147$$
$$\log p_{mm}\ (\text{nickel}) = -21{,}030/T + 9{\cdot}689$$

Heats of Adsorption on Nickel Films

D. F. KLEMPERER and F. S. STONE: 'Heats of Adsorption on Evaporated Nickel Films.'

Proc. Royal Soc., Ser. A, 1958, vol. 243, Jan. 14, pp. 375–99.

Report on study of the adsorption of oxygen, hydrogen and carbon monoxide on evaporated films of nickel prepared under various conditions. The specific aim of the research was to define more precisely the differences in behaviour which have been observed between nickel films and nickel powder. The conclusions drawn by the authors are correlated with earlier literature.

Oxidation of Nickel, Cobalt and Copper

J. J. CHESSICK, Y. F. YU and A. C. ZETTLEMOYER: 'The Oxidation of Nickel, Cobalt and Copper at −78°, −22°, 0° and 26°C.'

Preprints Proc. Second International Congress of Surface Activity, 1957, vol. 3, pp. DD 175–85.

The results of previous work on the oxidation of cobalt had been found to be at variance with theories advanced by Cabrera and Mott for the growth of very thin oxide films.

The investigation reported in this paper was therefore designed to study, by gas-adsorption and calorimetric techniques, the oxidation and regeneration of nickel, cobalt and copper powders at oxide-film thicknesses up to 30 Å and at temperatures in the range +26° to −78°C.

It was found that during the first oxidation at each test temperature multiple oxide layers formed in the course of an arbitrarily set time limit of 100 minutes. The bulk of the oxide, particularly in the case of the nickel and cobalt, formed by a fast, non-activated process. This reaction was followed by a slow process, which began abruptly, and in this case the amount of oxygen adsorbed was an exponential function of time.

The oxide surface layers on the nickel and cobalt specimens, and, to a lesser extent, those on copper, could be regenerated for further oxidation by heating in vacuo. Multiple oxide layers formed also on the regenerated samples, but to a lesser degree than during initial oxidation, and the amount of oxygen taken up decreased as the thickness of the oxide film increased. The fast and slow stages of oxygen adsorption were observed also in successive oxidations of the regenerated samples.

The authors propose a mechanism for the regeneration process, based on the formation of metal-lattice vacancies and their elimination by heating. The slow process of oxidation is discussed in terms of relevant work and theories in the literature.

Absorptiometric Determination of Trace Amounts of Nickel and Other Elements

J. Y. McALOREN and G. F. REYNOLDS: 'The Absorptiometric Determination of Traces of Chromium in Nickel and Vanadium, of Vanadium in Chromium and of Nickel in Chromium and Vanadium.'

Metallurgia, 1958, vol. 57, Jan., pp. 52–6.

In the laboratories of the Chemical Inspectorate, Ministry of Supply, it was recently found necessary to analyze pellets of chromic oxide containing 1 per cent. of vanadium pentoxide and pellets of mixed nickel and vanadium oxides, to determine whether interdiffusion of the metals had occurred as a result of heating the different pellets in contact with each other. Complete examination of the pellets required three procedures: estimation of traces of chromium in the presence of nickel and vanadium, vanadium when present with chromium, and traces of nickel in the presence of chromium and vanadium. The authors of this paper make a detailed report of techniques developed for such determinations. Results of typical analyses made by the methods recommended show good agreement with those obtained spectrographically.

Determination of Traces of Nickel and Other Elements in High-Purity Iron

E. HEFFELFINGER, D. L. CHASE, G. W. P. RENGSTORFF and W. M. HENRY: 'Analysis of High-Purity Iron.'

Analytical Chemistry, 1958, vol. 30, Jan., pp. 112–14.

The paper describes methods used in the laboratories of Battelle Memorial Institute. A process involving separation of metallic impurities by ether extraction of iron, and spectrographic analysis of the remaining aqueous solution, is used for estimation of manganese, copper, nickel, chromium, vanadium, cobalt, aluminium, lead, titanium, tungsten and zirconium. Sulphide precipitation and subsequent spectrographic analysis are employed for elements in the sulphide group. Direct arcing of ferric chloride is used for determination of silicon, magnesium and calcium. Detection limits, using these procedures, vary from 0·1 to 10 p.p.m. Boron is determined by standard spectrographic methods. For carbon a combustion technique is used: sulphur and phosphorus are determined spectrophotometrically. Determination of oxygen, hydrogen and nitrogen is by vacuum-fusion analysis.

Determination of Nickel in Plating Solutions

See abstract on p. 74.

Surface Analysis of Nickel Powder by Determination of Gas Adsorption

M. W. ROBERTS and K. W. SYKES: 'A Method of Surface Analysis and its Application to Reduced Nickel Powder.'

Proc. Royal Soc., Ser. A, 1957, vol. 242, Nov. 19, pp. 534–43.

The paper reports investigations on the possibility

of analysing the complex surfaces of reduced metals by comparing their adsorptive properties with the known behaviour of evaporated films.

Nickel prepared by reduction of the oxide in hydrogen at 450°C. for periods up to 12 hours is shown, by measurement of adsorption of hydrogen and krypton at −183°C., to have a surface containing up to 37 per cent. of clean nickel atoms. (Traces of silica introduced during preparation of the powder may account for the remainder of the surface.) Adsorption of hydrogen at −183°C. is effectively complete within ½ minute at about 10^{-5} mm. and all gas is desorbed by evacuation at 450°C. It is considered that oxide is probably not a significant impurity, because the powder does not exhibit the slow adsorption at 20°C. and above which is characteristic of incompletely reduced nickel. Such activated adsorption can be restored by addition of oxygen and eliminated again by further reduction. Its stoichiometry is considered to suggest that the process consists of hydroxyl formation.

Reduction of the sulphide gives nickel of appreciable surface area, but completely inert to hydrogen: it is presumed that a stable layer of sulphide is retained.

Some thermodynamic and kinetic problems involved in the preparation of metal powders having surfaces of known purity are briefly discussed, in the light of the experimental results reported.

ELECTRODEPOSITION AND OTHER COATING METHODS

Throwing Power in Bright-Nickel-Plating Solutions

P. MEULDIJK: 'Throwing Power in Efco-Udylite Bright Nickel Solutions.'

Metal Finishing Jnl., 1957, vol. 3, Dec., pp. 491–2, 494.

The aim of this paper is essentially practical: to report the influence of varying conditions on the distribution of nickel deposits made from commercial organic-brightened nickel electrolytes. Some dull-nickel-plating experiments were also made, for comparison.

Tests were carried out in a rectangular cell 250 × 75 × 80 mm. The two cathodes were of brass sheet and the exposed area on each was 48 sq. cm. They were so arranged that one of them was four times as far distant from the anode as was the other. The throwing power (T value) was measured as the percentage ratio of the weight of deposit on the cathode furthest away from the anode to the weight of deposit on the other cathode. A graph was drawn up according to the following formula:

$$T = \frac{A}{B} \times 100$$

in which

T = factor indicating throwing power of solution
A = weight of deposit (mg.) on furthest cathode
B = weight of deposit (mg.) on nearest cathode

(1) The first series of tests dealt with three dull-nickel solutions, a dilute type, a sulphate type and a high-chloride solution, all without any addition agents. Experiments were made to determine the influence of composition of the solutions and of current density and, in some solutions, the effect of temperature.

In all cases the high-chloride solution gave a better T value: changes in temperature or pH caused relatively small variation. In the high-chloride solution it is possible to obtain very high T values at low current density.

(2) In the second group of experiments various Udylite (organic) brighteners were added, in various concentrations, to the sulphate and the chloride solutions.

The presence of the brighteners effected no sharp change in T value, but there was a general tendency for throwing power to decrease. This was particularly noticeable in solutions containing No. 514 brightener.

(3) In the third group a study was made of the influence of various contaminants, on the T value of Nos. 514 and 514H solutions. The impurities added were zinc, copper, lead (each added in amounts of 25–50 and 100 mg/L), chromic acid (5, 10, 25 mg/L) and stearic acid. Lead caused a definite decrease in T value; the other metallic contaminants had little effect. Chromic acid lowered the T value to a marked extent; stearic acid had only a slightly unfavourable effect.

Nickel Plating of Aluminium

J. C. WITHERS and P. E. RITT: 'Firm Adherent Plating for Aluminum.'

Metal Finishing, 1958, vol. 56, Jan., pp. 53–4, 57.

The authors report experiments made with the zincate method (in two modifications) and with a method involving etching the aluminium base in a solution containing dextrin, water and trichloracetic acid, rinsing in acetone, and plating from a nickel-sulphate solution. Although the second method gave more satisfactory results than the zincate process, the adhesion obtained was not considered to be optimum, and further experiments were carried out to ascertain the possibility of improving it by fusing the deposited metal with the aluminium base. As a result of this work, the following treatment was developed:

1. Degreasing (vapour degreasing or soaking the aluminium in any suitable solvent).
2. Etching Treatment:
   (*a*) Etching in a 10 per cent. by volume solution of hydrofluoric acid (48-52 per cent.) for 10-15 seconds.
   (*b*) Rinsing.
   (*c*) Etching in 50 per cent. hydrochloric acid containing 9-10 g./L. of manganous sulphate ($MnSO_4.H_2O$), for 10-80 seconds, depending on the nature of the alloy.
   For the light alloys of aluminium, 20-30 seconds is usually sufficient: for some die- and sand-castings a more severe etchant may be necessary, e.g., 3 parts of nitric acid + 1 part of hydrofluoric acid.
   (*d*) Rinsing.

3. Nickel Plating, from a solution of the composition shown below:

	oz./gal.	g./L.*
Nickel sulphate ($NiSO_4.6H_2O$) ..	19	142
Magnesium sulphate ($MgSO_4.7H_2O$)	10	75
Ammonium chloride	2	15
Boric acid	2	15

Plating for about 10 minutes at 15 amp./sq. ft. gives the required thickness of nickel, 0·0001-0·0005 in. (0·000025-0·000127 mm.).

4. Heating at 800°-1050°F. (425°-565°C.).
(This treatment forms 'islands' of diffused nickel-aluminium, and other metals can be satisfactorily deposited on this surface. Copper, chromium, nickel, zinc, cadmium, gold, tin and solder are mentioned as having been deposited.

Adhesion tests (heating, bending and atmospheric storage) have shown no blistering, peeling or other indication of faulty adhesion on specimens plated by the procedure described.

Corrosion-Resisting Electrodeposited Coatings

J. ELZE: 'Protection from Corrosion by Means of Electrodeposited Coatings.'

Metall, 1958, vol. 12, Jan., pp. 320–8.

The author briefly surveys the three main functions of electrodeposited coatings: protection against corrosion, decorative effect, production of specific mechanical properties. He then discusses the broad technical and economic considerations affecting selection of the type of metallic coating to be used in specific applications. Attention is directed, *inter alia*, to the importance of giving due consideration to the chemical inter-relation between the basis and the coating metals and to the significance of coupling of metals in service.

The thicknesses necessary for corrosion-resisting coatings of various types are discussed and relevant German standards are listed, see below:

D.I.N. 50 961 Zinc Coatings on Steels
D.I.N. 50 962 Cadmium Coatings on Steel
D.I.N. 50 963 Nickel and Nickel/Chromium Coatings on Steel
D.I.N. 50 964 Copper/Nickel and Copper/Nickel/Chromium Coatings on Zinc and Zinc Alloys

Some comparison is made between German and American specifications for electrodeposited coatings.

The paper ends with critical discussion of the general corrosion-resisting characteristics of various types of coating, in relation to the applications for which they have proved suitable. The importance of freedom from porosity is emphasized as a significant factor determining protective value.

Electroless Deposition of Nickel

C. H. DE MINJER and A. BRENNER: 'Studies on Electroless Nickel Plating.'

Plating, 1957, vol. 44, Dec., pp. 1297–305.

Since details of the electroless plating process were first published, in 1946 by Brenner and Riddell, investigation has been directed mainly to development of its practical application. Studies of fundamental factors involved in the process have been relatively rare, and the work now reported was undertaken to fill in some of the gaps in basic knowledge of the variables affecting deposition by this method. Particular attention was given to factors which influence rate of deposition of metal.

The compositions of the solutions used and of the operating conditions employed are given in Table I, page 72. Solution 2 (designated the Hydrac bath) was used as control for evaluating the characteristics of the other solutions. Low-carbon steel was the basis material.

The test programme was arranged to secure information on the following variables:

(1) *Influence of pH on Rate of Deposition*

It was known from the literature that the rate of deposition from the acid electroless solution accelerated with increase in pH value, but the optimum value for solutions used in continuous deposition had not been clearly established. Use of a high-pH solution, to take advantage of the faster rate of deposition, is impracticable because (*a*) at a value as high as 6 hydrogen ion is formed during the plating reaction, and (*b*) solubility of nickel phosphite decreases with increase in pH value, resulting in precipitation of the phosphite, which initiates decomposition of the solution. The experimental work reported covered determination of the effect of variation in pH, on rate of deposition of nickel from solutions 2, 3, 4 and 5.

(2) *Influence of the Type of Organic Acid used, on Rate of Deposition*

Previous work, by Brenner and Riddell, had revealed that deposition of electroless nickel was affected by the type of organic acid incorporated in the solution. In the present work this effect was studied in detail, using hydroxyacetic, lactic, acetic, citric, succinic, tartaric, phthalic, salicylic and substituted-acetic acids. Maximum rates of deposition obtainable with the respective acids, at various concentrations, were compared, and the effect of the acids on the stability of the solutions was observed.

(3) *Precipitate Formed in Electroless Plating Solutions*

A study was made of the nature of the green precipitate which forms after a period of plating, initiating nucleation of nickel and causing spontaneous decomposition of the solution. Analysis indicated that the precipitate is probably a mixture of two nickel phosphites, $NiHPO_3.H_2O$ and $NaNiPO_3$. Both the concentration of phosphite present and the pH value of the solution influence the solubility of nickel phosphite in the solution, and since satisfactory operation is dependent on optimum solubility, a study was made of onset of precipitation of the salt at boiling temperature, in solutions of varying phosphite concentration and pH. Development of turbidity after boiling for 30 minutes was taken as indication of saturation with nickel phosphite.

* Approximate conversion.

Table I

Compositions of Electroless-Plating Solutions

Constituents

(g./L.)

Solution	$NiCl_2.6H_2O$	$NaH_2PO_2.H_2O$	Hydroxy-acetic Acid	Succinic Acid	Sodium Citrate	Sodium Acetate	NH_4Cl
(1) Inorganic	30	10	—	—	—	—	—
(2) Hydroxyacetic ('Hydrac' solution)	30	10	39	—	—	—	—
(3) Low-Hydroxyacetic ('Low-Hydrac' solution)	30	10	25	—	—	—	—
(4) Citrate	30	10	—	—	15	—	—
(5) Citrate-Acetate ..	30	10	—	—	15	5	—
(6) Succinate	22	30	—	15	—	—	—
(7) Alkaline	30	10	—	—	100	—	50
(8) Alkaline with wetting agent	30	10	—	—	100	—	50

Operating temperature .. 95°-100°C.

pH { Acid-type baths .. .. 4·0-4·3, adjusted with sodium hydroxide
Alkaline-type baths .. 8·5-9·5, „ „ ammonium hydroxide

(4) *Effect of Iron or Copper Salts on Operation of the Solution*

The electroless plating solutions are very sensitive to impurities, and there has been some suspicion that copper and iron present in some of the compounds used may be responsible for low rates of plating and instability of the baths. Effect of such contamination was studied by addition of ferrous chloride (0·5 g./L.) or of $CuSO_4.5H_2O$ (4 g./L.) to the Hydrac solution.

(5) *Reactivation of Spent Solutions*

In the plating process hypophosphite is consumed, and to maintain continuous operation it is necessary to add a solution of hypophosphite at intervals. The authors attempted to rejuvenate the solutions by electrolytically reducing phosphite to hypophosphite, but the results were not successful. It is considered, however, that there may still be a possibility of developing an electrolytic method of rejuvenation.

(6) *Effect of Inhibitors and Other Substances, on Rate of Deposition*

Several hypotheses have been advanced to explain the mechanism of electroless deposition: they usually incorporate the idea of initial discharge of hydrogen which, in the presence of nickel as catalyst, reduces nickel ion to nickel. Brenner has proposed a slightly different hypothesis, involving a two-stage process. First there is catalytic decomposition of hypophosphite ion, leading to discharge of hydrogen, whilst the second step involves a transfer of energy from the discharging hydrogen ion to the nickel ion, thereby activating it so that it can react with the hypophosphite ion. On the basis of this hypothesis, the rate and manner in which hydrogen is discharged in step 1 is significant in determining rate and efficiency of deposition of nickel in step 2. In this connexion a study was made of the effect, on electroless plating, of substances known to influence hydrogen overvoltage: selenic acid, thiourea, potassium thiocyanate, peptone, pyridine, cadmium chloride and benzene disulphonic acid. The low-Hydrac solution No. 3 was used for these experiments.

(7) *Protective Value of Electroless Nickel, Electrodeposited Nickel and Electrodeposited Nickel-Phosphorus Alloys*

It has been claimed, mainly on the basis of salt-spray tests, that electroless nickel affords better protection against corrosion than is obtained from electrodeposited nickel coatings. To test the validity of this claim, as affecting behaviour in atmospheric conditions, plated panels of steel were exposed in two locations, Kure Beach (marine) and Washington (industrial) atmospheres. The electrodeposited nickel coatings were made from a conventional all-chloride bath; the electrodeposited nickel-phosphorus coatings were representative of two levels of phosphorus content (3 and 9 per cent.), and the four solutions used

for preparation of the electroless nickel specimens included acid and alkaline types and one in which a wetting agent was added to lessen pitting. In all cases two thicknesses of coating were deposited, 0·5 and 1 mil. The behaviour of the coatings was rated at intervals of a few months; total exposure period was 14–15 months. Results are compared with those of salt-spray tests carried out on duplicate specimens.

From the detailed data reported for the individual series of experiments, the authors draw the following general conclusions:

(1) Solutions containing hydroxyacetic acid gave the highest rate of deposition. Of these, solution No. 3 gave the best results: it was less sensitive to pH and yielded brighter deposits. In this bath, however, the solubility of nickel phosphite was lower and this factor shortened the life of the solution.

(2) Increase in amount of organic acid present caused gradual increase in rate of deposition of nickel up to a maximum, followed by decrease. Of the organic acids studied, the lactic acid gave the highest rate, and hydroxyacetic acid was the next most effective. With succinic acid the rate of deposition of nickel was slightly lower than that obtained with the hydroxyacetic solution.

(3) Solubility of nickel phosphite decreased with increase in pH, resulting in precipitation of phosphite and decomposition of the solution. It is recommended that an acid bath should be operated at pH between 4 and 4·5. At a given pH solutions containing no organic acid dissolve less salt than those in which organic acids are present.

(4) Iron or copper salts, in the amounts likely to be present, are not seriously detrimental to the operation of the electroless solutions.

(5) Some inhibitors, when present in very small concentrations (0·1–4 mg./L.) appreciably increased the rate of deposition; at slightly higher concentrations they slowed it down and at still higher concentrations they entirely prevented deposition of metal. Examples of such inhibitors are thiourea, potassium thiocyanate and selenic acid. Thiourea considerably increased the rate of deposition and also improved the brightness of the deposit and the stability of the bath. None of the addition agents used deleteriously affected the stability of the solution.

(6) Coatings of nickel-phosphorus alloys containing 8–10 per cent. phosphorus, whether deposited electrolytically or by the electroless process from an acid solution, protected steel against rusting better than did electrodeposited nickel coatings. The tarnish-resistance of these alloy coatings was better than that of normal dull-nickel coatings or of nickel-phosphorus coatings of low phosphorus content. The protective properties of electroless coatings made from alkaline baths were inferior to those deposited from acid solutions.

Sulphur-Dioxide Test for Evaluation of Electrodeposited Coatings

J. EDWARDS: 'Accelerated Corrosion Testing of Chromium-Plated Articles: Sulphur-Dioxide Test.' *Electroplating and Metal Finishing*, 1958, vol. 11, Jan., pp. 17–22.

Abstract of paper presented to joint meeting of the Institute of Metal Finishing and the Society of Chemical Industry, in London, October 1957.

See also full form of paper in *Trans. Inst. Metal Finishing*, 1958, vol. 35, pp. 55-78: *Bull. Inst. Metal Finishing*, 1957–58, vol. 7, No. 4.

The material had previously been issued to members of the British Non-Ferrous Metals Research Association as Research Report A.1143.

The author emphasizes the difficulty experienced in evolving accelerated tests which will give results comparable with service behaviour, and urges the futility of tests which fail to give such indication. The limitations of various accelerated tests which have been proposed are briefly reviewed. The fact that the majority of plated components are used in industrial atmospheres, in which the corrosive medium of obvious significance is sulphur dioxide, suggested the value of SO_2 as a test corrodent. In this paper details are given of investigations carried out by the British Non-Ferrous Metals Research Association with that medium.

Preliminary work indicated that a sulphur-dioxide test originally devised for evaluation of tin-alloy coatings offered most promise. In this test samples are suspended, at room temperature, in a closed vessel containing SO_2 solution. This test had none of the disadvantages of other tests which had employed SO_2: the sulphur-dioxide concentration could be well controlled, and moisture collected on the surface of the specimens only at the sites of attack. The pattern of the attack was therefore not obscured by spread of corrosion products over the surface.

Under this test components plated with nickel/chromium, copper/nickel/chromium, copper/brass/chromium, and copper/tin-nickel showed good correlation with similar specimens subjected to outdoor exposure. The agreement was better than was obtained from similar experiments carried out by the acetic-acid/salt-spray test, both with regard to the degree of protection conferred by the deposits and the type of attack undergone.

Details are given of check tests in which nine members of the British Non-Ferrous Metals Research Association co-operated. In one series the specimens used consisted of nave plates coated with nickel/chromium or copper/nickel/chromium. Each plate was cut into four sections: three were distributed to various laboratories for testing by the sulphur-dioxide procedure and the fourth was exposed for six weeks in an industrial atmosphere. The equipment used in the different laboratories varied considerably, but the SO_2 concentration was in all cases 1 vol. per cent. and humidity was maintained just below 100 per cent. The specimens were tested for 24 or 96 hours, and after test were re-assembled

to enable correlation of results. (Reproducibility obtained in the different laboratories is illustrated by photographs of the corroded discs.) Allowing for certain variations in test conditions, the results showed satisfactory agreement, and it is concluded that a 24-hour SO_2 test is sufficient to reveal the nature of the attack likely to develop in a short-term exposure test: extension to 96 hours appears to be unnecessarily severe for most purposes, although in work concerned with improving the durability of plated coatings it will be necessary to use periods longer than 24 hours.

Test conditions recommended are:

SO_2 concentration	0·5-2·0 vol. %
Humidity	95% to a little below 100%
Temperature	17°-25°C.

The results of all the tests made indicate that the sulphur-dioxide procedure reveals large discontinuities, such as cracks or holes (i.e., gross porosity) in (copper)/nickel/chromium deposits on steel, and is capable of developing a pattern of pitting corrosion similar to that produced by a service environment, but that the results are dependent to some extent on the thickness of the nickel or copper/nickel undercoat and of the chromium. In addition, the SO_2 test reveals incomplete coverage by chromium and cracks within the chromium. The authors suggest that the test may be applicable also to nickel/chromium-plated brass and copper/nickel/chromium-plated zinc-alloy die castings.

It is emphasized that further work is necessary before the test can be regarded as an absolute criterion for rejection or acceptance of plated components.

See also

Evaluation of the Acetic-Acid/Salt-Spray and Sulphur-Dioxide Tests

'Two New Accelerated Corrosion Tests: An Assessment',
ibid., pp. 15-16.

This article is to be read in conjunction with the paper abstracted above and the report by HOOPER which critically reviewed the performance and sphere of application of the acetic-acid/salt-spray test (*ibid.*, 1957, vol. 10, Dec., pp. 403-8; *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 44). This article attempts to assess the value and future position of these two tests, on the basis of information available to date.

After a brief résumé of the apparatus and conditions employed, the advantages and limitations of the two methods are considered, as assessed (1) by their ability to indicate the protection conferred by various types of coating, and (2) by their suitability for use as acceptance tests ensuring compliance with specifications.

A procedure to be used as an acceptance test is not necessarily required to give a result entirely similar to that which would be obtained under longer-term atmospheric exposure. The main requirement in this type of test is rather that it should show up clearly and rapidly whether the coating has any defects likely to cause failure in service. It is concluded by the writer that for this latter purpose the SO_2 test is the most satisfactory one so far devised for examination of nickel/chromium coatings on steel. Since moist SO_2 will attack nickel, the test is not satisfactory for testing nickel coatings which have no chromium overlay. If a satisfactory chromium coating has been applied, the plating will normally withstand the SO_2 test for 24 hours, assuming that the nickel coating is satisfactory. If, however, the chromium coating has many cracks the nickel will be corroded at those points and the cracks will show up as characteristic green lines on the surface of the article. This crack pattern is very similar to that produced in an industrial atmosphere. If the nickel deposit is of poor quality it is penetrated, and rust spots are clearly visible above the green crack pattern. Such spots indicate that the coating is unsatisfactory. It is thus possible, if the test is interpreted by a skilled observer, to determine (*a*) whether the nickel undercoat is satisfactory, and (*b*) whether the chromium deposit itself is good or bad.

For plated zinc-base die castings the acetic-acid/salt-spray test is probably the more satisfactory, but no entirely satisfactory procedure has yet been devised for coatings on brass and other copper-base alloys.

In summary, it is concluded that both the tests have useful applications and that both will find a place in industry.

Determination of Nickel and Boron in Plating Solutions

D. E. FORNWALT: 'A Flame Spectrophotometric Method for the Determination of Nickel and Boron in Plating Solutions.'
Analytica Chimica Acta, 1957, vol. 17, Dec., pp. 597-603.

In 1955 DEAN and THOMPSON made a study of the flame photometric determination of boron in 1 : 1 methanol-water solution, using the boron-oxide band system (*Anal. Chem.*, 1955, vol. 27, p. 42). The present authors studied the application of this method to the determination of both boron and nickel. Their results led to the conclusion that the procedure offers a rapid and accurate means for determination of the two elements in plating solutions and that it may be readily adaptable to their determination in other solutions.

NON-FERROUS ALLOYS

Extraction of Nickel and Cobalt: Bibliography

See abstract on p. 68.

Copper and Copper Alloys: Literature Review

E. VOCE: 'Copper and Copper Alloys. A Survey of Technical Progress during 1957.'
Metallurgia, 1958, vol. 57, Jan., pp. 3-15.

Highly condensed but informative review of progress,

culled from published literature, to which reference is given in a *bibliography* of 309 items. The survey is made in sections relating, respectively, to production of copper, foundry practice, fabrication, plating and finishing, properties and applications, corrosion, joining, powder metallurgy, and inspection, testing and analysis. The items to which attention is directed include information on alloys in which nickel is used in conjunction with copper.

Fracture Characteristics of Copper-base Alloys

N. C. HOWELLS and E. A. LANGE: 'Fracture Characteristics of Copper-base Alloys.'

U.S. Naval Research Laboratory N.R.L. Report 4925, Apr. 17, 1957; 11 pp. *P.B.* 121933.

The now considerable literature on brittle failure of steel structures has emphasized the significance of temperature, and the temperature at which the energy level of 10 ft.-lb. is reached in the impact test has been widely accepted as the point corresponding to the ductile→brittle condition in steels.

The investigation described in this report was initiated to determine whether the impact energy of brasses and bronzes commonly used by the U.S. Navy would similarly decrease with fall in temperature and whether any of these alloys would become susceptible to brittle fracture at Charpy V levels at or below 10 ft.-lb. The 15 alloys tested were representative of gunmetal, M bronze, leaded bronze, tin-nickel bronze, phosphor bronze, silicon-brass and -bronze, low- and high-tensile manganese bronze, and nickel-aluminium bronze.

Results of drop-weight tests and Charpy V-notch tests, at temperatures between 210° and −300°F. (99° and −184°C.), show that the fracture relationships of copper-base alloys differ from those of steel, in that a Charpy V energy level of 10 ft.-lb. does not indicate a brittle condition in the copper-base alloys. Of the materials examined, only an alloy which gave an abnormally low tensile elongation value (less than 2 per cent.) fractured in a brittle manner. It is noted, however, that the ductility of high-tensile manganese bronze becomes very low at temperatures below −100°F. (−73°C.). The interrelationships between impact energy, tensile elongation and notch ductility for the alloys are discussed.

Electrical Conductivity and Hall Constant of Copper-Nickel and Copper-Nickel-Zinc Alloys

W. KÖSTER and W. SCHÜLE: 'Electrical Conductivity and Hall Constant. IV. Copper-Nickel Alloys; V. Copper-Nickel-Zinc Alloys.'

Zeitsch. f. Metallkunde, 1957, vol. 48, Nov., pp. 592–4; 595–600 (see also related work, *ibid.*, 1957, vol. 48, Sept., pp. 485–95).

I. The experimental investigations covered binary alloys containing 15, 20, 25, 30 and 45 per cent. of nickel. Electrical resistivity and Hall constant were studied on strip specimens, as a function of mechanical and thermal treatment.

II. Study of a range of copper-zinc-base alloys, in which there was gradual replacement of zinc by nickel: the broad ranges covered fell within the limits

Copper + 25% zinc to copper + 30% nickel
„ + 35% „ „ „ + 50% „

Ductile Chromium and Chromium-base Alloys

See abstract on p. 82.

Symposium on Structural Sandwich Design and Materials

See abstract on p. 78.

Nomenclature of Alloy Phases

AMER. SOC. TESTING MATERIALS: 'What Can be Done to Improve Alloy Phase Nomenclature ?'

A.S.T.M. Bull., 1957, No. 226, Dec., pp. 27–30; *T.P.* 215.

During the past few years A.S.T.M. Committee E-4 on Metallography has been engaged in developing proposals for an improved nomenclature for metallic phases. This report contains proposals which have been worked out by members of the Committee, in consultation with experts in various countries.

The paper opens with brief discussion of some of the main objections to systems of nomenclature at present used, and of contradictions to which such usage gives rise. In view of the growing complexity of the alloys which are currently being developed, this matter is becoming increasingly important.

It is suggested that the most likely basis upon which a self-consistent systematic nomenclature might be built is crystal structure, and the method of designation now proposed is founded on this principle. The proposals are being submitted to leading metallurgical societies throughout the world, with an invitation for constructive criticism or ideas for alternative systems.

An appendix contains comments, by F. N. RHINES, on the Committee's proposals, and suggestions for another system which he considers preferable.

Electrical Resistivity of Nickel-Palladium Alloys

A. I. SCHINDLER, R. J. SMITH and E. I. SALKOVITZ: 'Electrical Resistivity of the Ni-Pd Alloy System between 300°K. and 730°K.'

Physical Rev., 1957, vol. 108, Nov. 15, pp. 921–3.

Electrical-resistivity measurements reported in 1956 had shown that resistivity reaches a maximum at approximately 70 at. per cent. palladium, at 4·2°, 77° and 300°K. (−269°, −196° and +27°C.).

The present paper reports measurements on the same alloy system from room temperature to 730°K. (457°C.). The maximum in the resistivity value was found to shift from 70 at. per cent. palladium at room temperature to the 50-50 composition at temperatures at which all the specimens are paramagnetic. At these elevated temperatures, it was found that Matthiessen's rule is obeyed over the entire range of composition. The general behaviour of the alloys may be interpreted in terms of the dependence of *s-d* scattering upon temperature and composition.

Constitution of Alloys: Literature Survey 1956

H. F. SPENGLER: 'Recent Progress in Research on the Constitution of Alloys.'

Metall, 1958, vol. 12, Feb., pp. 105–13.

This review represents a departure from previous papers in the series, which have comprised classified but unannotated references to recent literature (see, for example, *Nickel Bulletin*, 1957, vol. 30, No. 3, p. 38). Attention is now directed to the appearance, in 1956, of the revised edition of the Institute of Metals Monograph and Report No. 2 covering 'The Constitutional Diagrams of Alloys'. That publication will be constantly revised, and the present author considers that his yearly lists will therefore be unnecessary. He is proposing in future to issue commentaries on the main results of the previous year's work. The present review is based on a *bibliography* of 109 items. Among the systems on which comment is made are the following nickel-containing types: Hg-Ni, Ni-Pu, Ni-Te, Al-Cr-Ni, Al-Ni-Ti, C-Ni-Ti, Cr-Ni-Ti, Fe-Ni-V, Mn-Ni-V, Al-Cr-Ni-Ti, Al-Cr-Fe-Ni-Ti, Al-Cr-Ni-Fe-W.

Resistance of Nickel-containing Aluminium Alloys to High-Temperature Water

See abstract on p. 87.

Resistance of Metals to Corrosion by Cement-Water Mixture

See abstract on p. 88.

X-Ray-Diffraction Measurement of Stress in Monel Welds

E. H. KINELSKI and J. A. BERGER: 'X-Ray Diffraction for Residual Stress Measurements of Restrained Weldments.'

Welding Jnl., 1957, vol. 36, Dec., pp. 513s–17s.

Although the use of X-ray techniques for examination of metals has rapidly increased during the past 20 to 30 years, very little use has been made of such methods in the welding field. The authors of this paper urge the value of examination by X-ray diffraction for this purpose, supporting its claims by results on Monel welds. The report of experimental work is introduced by discussion of the theory of X-ray diffraction and its application to the study of stress distribution.

In the experiments described, the parent metal was ½-in. thick Monel (nickel-copper alloy containing small amounts of iron and manganese). A simple butt-weld specimen was chosen to illustrate the usefulness of the method in measuring residual stresses resulting from welding under restraint. Three commonly used welding procedures were employed:

(i) metallic-arc, using a flux-coated electrode.
(ii) gas-shielded, tungsten-arc, using a bare filler wire.
(iii) gas-shielded, consumable-electrode, using a bare filler wire.

Although the alloy used does not normally require pre-heat or the maintenance of high interbead temperature, a large number of complex alloy systems would require such treatment, where high stresses might be anticipated. The welds were therefore made first with no pre-heat and an interbead temperature of 100°F. (38°C.), and second with pre-heat at 300°F. (150°C.) and an interbead temperature of the same level. The resulting stress data were graphically analyzed and were compared with the behaviour which could be anticipated from the welding processes used, with their variable heat inputs.

The results indicate that only in the case of consumable-electrode welds has the 300°F. (150°C.) pre-heat and interbead temperature a beneficial effect in reducing the stress gradient.

The X-ray-diffraction method proved very satisfactory as a means of studying the effect of welding variables.

Nickel-Aluminium-Bronze Marine Propellers

'Alloy Builds a Better Wheel.'

Reprint from *Motor Boating*.

Issued by INTERNATIONAL NICKEL CO., INC., 1957; 4 pp.

The article discusses the advantages derived from the use, for marine propellers, of Ni-Bral, a nickel-aluminium bronze developed by The International Nickel Co., Inc. The article is of particular interest when read in conjunction with the paper by HUDSON (*Shipbuilder*, 1957, vol. 64, Dec., pp. 681–7; *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 46), since it gives further details of operational experience with propellers made from a type of alloy described in that paper.

The greater strength conferred by use of the nickel-aluminium-bronze alloy permits 10 per cent. reduction in thickness of the blades as compared with those made in the conventional manganese-bronze alloy. The general improvement in operational efficiency, together with the high resistance of the alloy to corrosion, impact and fatigue, make the material ideal for the propellers of many types of ship. Its advantages and superior service behaviour, *vis-à-vis* manganese bronze, are indicated in this article by specific examples of performance in small motor cruisers, pleasure boats, high-speed racing boats and other craft. The alloy has been approved for use in the propellers of U.S. Navy and coast-guard ships. One of the propeller manufacturers comments that five-bladed propellers became feasible only by taking advantage of the properties offered by the new material. An economical design required narrower and thinner blades than those usual in propellers of manganese bronze, and it was therefore necessary to have a stronger alloy. Nickel-aluminium bronze met this requirement.

Nickel-Aluminium-Bronze Pump Parts

'Ni-Al Bronze Protects Pumps from Corrosion.'

INCO Nickel Topics, 1958, vol. 11, No. 1, p. 5.

In the use of nickel-aluminium bronze for marine propellers, advantage has been taken of, *inter alia*, the high resistance of the alloy to corrosion by sea

water (see previous abstract). This note gives details of another application which makes use of this property; use as a material of construction for the fluid ends of pumps handling highly corrosive salt water in oilfield waterflood service. After two to three years' service the nickel-aluminium-bronze components have shown no signs of corrosion, a performance which is greatly superior to that of the carbon steel previously used.

Nickel-Alloy Springs

See abstract on p. 86.

Absorptiometric Determination of Trace Amounts of Nickel and Other Elements

See abstract on p. 69.

CAST IRON

Surface Hardenability of Cast Irons

H. SCHIFFERS, D. AMMANN and E. BRUGGER: 'Surface Hardenability of Various Types of Cast Iron.'
Giesserei, 1957, vol. 44, Sept. 26, pp. 583–8.

The authors describe apparatus and technique by which the principle of the Jominy quench test can be applied to study of the depth of hardening obtained by flame treatment and water cooling (quenching) of rotating specimens. Comparison is made with hardenability tests carried out by the normal Jominy procedure on furnace-heated specimens, and equipment used in both series of tests is illustrated.

In order to study the influence of composition on surface hardenability, tests were made on several types of cast iron: unalloyed grey iron, spheroidal-graphite iron made by the nickel–magnesium process, and low-alloy cast irons containing nickel, chromium, molybdenum, vanadium and titanium, in various percentages and combinations. Structural examination prior to hardening showed that all the irons except those alloyed with vanadium + titanium had an entirely pearlitic matrix. In the vanadium-titanium irons the matrix was ferritic-pearlitic.

Hardenability curves are shown for both the furnace-heated and the surface-hardened irons, giving hardening effects at intervals up to 80 mm. from the position of quenching. In most cases the data relate to the effects obtained by surface-hardening at 840°C., but the work included treatments at five temperatures within the range 780°–990°C., in order to study the effect of hardening temperature on depth of penetration. Data on a nickel-chromium-alloy cast iron are given as typical of the observations made in that connexion, and structural effects produced by the use of the various temperatures are illustrated.

With regard to the effect of composition and structure, the authors' conclusions are as follows:

The behaviour of the unalloyed irons confirms earlier conclusions drawn with reference to the influence of graphite distribution, degree of saturation and silicon content. The good response of spheroidal-graphite iron to surface hardening, previously reported by Timmerbeil, is also confirmed.

Nickel (0·45, 0·78 and 1·08 per cent.) had a progressively beneficial effect on hardenability, but no further improvement resulted from the presence of chromium up to 0·37 per cent. (the highest level studied). The marked effect of molybdenum, already well known in relation to steel, is confirmed by the results on the cast irons in which that element was present. The vanadium–titanium irons, in spite of their slightly lower initial hardness, showed a good degree of hardenability.

The final section of the paper reports investigation of the 'holing' effect which is sometimes liable to occur in surface-hardening of cast iron. This removal of small particles from the surface has been attributed by some investigators to burning-out of graphite, but the present authors disagree with that explanation, and suggest that it is a case of breaking-up and falling-out of small crystals of martensite which occur alongside of the graphite. Arguments in support of this view are advanced.

CONSTRUCTIONAL STEELS

Production of Cast Nickel-containing Constructional Steels for Low-Temperature Service

W. A. KOPPI: 'Some Aspects of Melting Low-Temperature High-Impact Steels.'
Amer. Inst. Mining, Metallurgical and Petroleum Engineers, Preprint, Dec. 1957; 7 pp.

The paper discusses factors calling for consideration in the manufacture of ferritic cast steels to A.S.T.M. Specification A.352-55T, which covers four grades of cast steel: carbon steel, a carbon-molybdenum steel, and two grades of nickel steel (containing, respectively, 2-3 and 3-4 per cent. of nickel). The specification requires that the steels retain an impact strength of 15 ft.-lb. at test temperatures, respectively, of −50°, −75°, −100° and −150°F. (−45°, −60°, −73° and −100°C.).

Methods of countering the effects of hydrogen, nitrogen and oxygen are discussed and particular reference is made to the avoidance of non-metallic inclusions which, accumulating along the grain boundaries, can lower impact strength. It is urged that close attention must be paid to deoxidation processes, since they govern, to a large extent, three factors which can affect the impact strength of steels: cleanliness of the metal, type of sulphide inclusion, and grain size. In this connexion the formation of sulphide inclusions is discussed and consideration is given to their effect on impact behaviour. The use of manganese and silicon is recommended as producing the cleanest steels; final deoxidation with

aluminium should be followed by additions of silicon, adjusted to meet the composition requirements for the individual steels.

As an illustration of the practical application of the general principles laid down, the author describes, step by step, the melting processes used at the Lebanon Steel Foundry, Lebanon Pa., U.S.A. The results obtainable by these methods are demonstrated by data on properties typical of the steels so made.

Vacuum Melting of Nickel-containing Steels

See abstract on p. 81.

Symposium on Structural Sandwich Design and Materials

AMER. SOC. TESTING MATERIALS: 'Symposium on Structural Sandwich Constructions.'
A.S.T.M. Special Tech. Publn. No. 201.
Published 1957; 103 pp.
Price: Members, 2 dollars; Non-Members, 2.75 dollars.

This volume contains the papers and discussion presented at the Second Pacific Area National Meeting of the Society, Sept. 1956. The Symposium was sponsored by Committee C-19, which was established to standardize sandwich construction and introduce test methods for such composite structures. Brief notes on some of the papers are given below.

T. P. PAJAK: 'Introduction', p. 1.
Remarks on the scope of the symposium and on the increasing recognition, by industry, of the value of sandwich structures.

J. R. BATTALORA and D. E. PULSIFER: 'High-Temperature Testing of Adhesives for Aircraft Structural Application',
pp. 3-11; disc., pp. 11-12.

Details are given of some tests which may be used to evaluate the lap shear strength and resistance to peeling of adhesives employed for metal-to-metal bonding and for bonding of honeycomb cores to metallic facings.

W. G. PLUMTREE and W. CHEORVAS: 'Effect of Dimensional Factors and Temperature on the Shear Strength of Aluminum Honeycomb',
pp. 13-21; disc., p. 22.

J. M. STEVENS: 'Sandwich in the Design of Helicopters',
pp. 23-31; disc., pp. 31-2.

Discussion of the advantages derived from use of sandwich structures in the rotor blades of helicopters. Details are given of the structural features of blades in which aluminium honeycomb-core materials have been incorporated. Stainless-steel strip is used to protect the leading edge of the blades from erosion.

M. L. SHERIDAN and H. R. MERRIMAN: 'Empirical Study of Bonded Joints for Sandwich Construction',
pp. 33-48; disc., p. 49.

The paper presents data accumulated over a period of years by a U.S. aircraft firm, on the behaviour of various types of adhesive-bonded joint, as judged by strength tests. The data are derived mainly from aluminium-base materials, but tests were carried out also on 17-7 P.H. and Type 301 chromium-nickel stainless steels. The strength of the joints is presented as a function of (1) the mechanical properties of the adherent metals, (2) the geometry of the joint, (3) the mechanical properties of the adhesives, and (4) environmental variables, such as time of loading and temperature conditions.

R. E. PARKINSON: 'Selection of Materials for Architectural Sandwich Panels',
pp. 50-4; disc., pp. 55-6.

The author reviews criteria by which materials may be selected for use in architectural 'curtain-wall' panels, pointing out how the requirements vary from those of primary importance in sandwich materials for aircraft. Properties desirable in 'architectural' panels are discussed and consideration is given to factors which affect choice of materials for facings, adhesives and cores. Materials recommended as suitable for panel facings are 18-8 chromium-nickel or chromium-manganese-nickel stainless steels, anodized aluminium, porcelain enamel on a steel or aluminium base, and glass or ceramics. Stainless steel is an excellent facing material where its natural metallic colour is acceptable, and lamination of thinner sheets to reinforce structural sandwiches may partially offset economic disadvantages. Recently porcelain has been applied to textured sheets of stainless steel, to give colour highlighted by the bare stainless steel.

R. G. STEELE and A. C. MARSHALL: 'Recent Developments in Sandwich Construction, including Heat-Resistant Materials',
pp. 57-62; disc., pp. 63-4.

The factors responsible for the increasing use of sandwich structures in airframes are briefly reviewed and the trend towards greater utilization of newly developed aluminium alloys as core materials is discussed. The major part of the paper, however, is given up to consideration of the problems confronting the design engineer as a result of the high temperatures to which aircraft structures are subjected in supersonic flight: in recent years the temperatures which such structures are required to withstand have risen from 260°F. to 1800°F. (125° to 980°C.).

The solution to the so-called 'heat-barrier' is being sought in the development of new, or adaptation of existing, materials and techniques for sandwich cores. Stainless steels, of the 18-8 and the age-hardenable 17-7 P.H. types, are, for example, already in service at temperatures up to 1000°F. (540°C.). The authors present data on the high-temperature

shear strength of standard aluminium and plastic core materials and also on the theoretically derived properties of high-temperature alloys which offer promise of development for such applications, at temperatures up to 1800°F. (980°C.). These include Inconel X, 17-7 and A-286 age-hardenable steels and 18-8 stainless steel. Reference is made to recently developed plastic honeycomb structures, modified adhesives for bonding, and improved methods for brazing sandwich structures intended for use at high temperatures. The expected temperature limits at which various materials will give satisfactory service as facing, bonding or core components are given. An appendix to the paper identifies broadly the nature of proprietary and other specially designated materials referred to in the text.

E. W. KUENZI: 'Methods of Testing Sandwich Constructions at Elevated Temperatures', pp. 65–72; disc., p. 72.

Illustrated description of apparatus for testing various properties of sandwich constructions at elevated temperatures: *inter alia*, details of tests to determine edgewise compressive strength and flat-wise flexural strength.

W. J. SNODDON: 'Metal-to-Resin Adhesion as Determined by a Stripping Test', pp. 73–82.

The method described, which involves curing the resin in contact with a metal foil and subsequently stripping the foil from the resin under controlled conditions, was applied to evaluation of the adhesion of epoxy resin to steel, aluminium and brass foils.

J. S. ARNOLD: 'An Ultrasonic Technique for Non-Destructive Evaluation of Metal-to-Metal Adhesive Bonds', pp. 83–92.

Details are given of the theory and mechanism of the STUB-meter (Standford ultrasonic bond meter). The oscilloscope patterns resulting from typical bonds are compared with the strengths of the specimens as determined by subsequent destructive tests.

R. E. ANDERSON: 'Non-Destructive Testing of Bonded Metal Sandwich Structure', pp. 93–102; disc., p. 103.

This paper was presented at the Session on Plastics, Sept. 1956. It is included in the present volume because of its close relationship to the subject of the Symposium. It describes ultrasonic apparatus, the 'Immerscope', which has been adapted to testing adhesive bonds in sandwich structures. Typical results obtained by means of the apparatus are given.

Corrosion of Structural Steels in Catalytic Reformers

See abstract on p. 88.

Temper Brittleness in Nickel-Chromium Steels

N. V. TOLSTOGUZOV and A. D. KRAMAROV: 'Effect of Phosphorus and Manganese on Temper Brittleness in Chromium-Nickel Steel.'

Metalloved. i Obrabotka Metal., 1957, No. 2, pp. 32–9.

The percentage chemical compositions of the steel studied were in the range carbon 0·30–0·45, silicon 0·11–0·51, manganese 0·07–0·75, nickel 2·47–3·45, chromium 1·02–1·69, molybdenum 0–0·15, phosphorus 0·005–0·09, and sulphur 0·005–0·019. Tests were made at room temperature and at temperatures down to −60°C. Results are summarized in *Chemical Abstracts*, 1957, vol. 51, Oct. 25, p. 15,368.

N. V. KAZAKOVA and N. V. KOROLEVA: 'Temper Brittleness of Steel.'

Metalloved. i Obrabotka Metal., 1957, No. 2, pp. 39–45.

Examination of low-alloy nickel-chromium steels, in the tough and brittle conditions, by means of electron-microscopical investigations and by Charpy impact tests. For results, see *Chemical Abstracts*, 1957, vol. 51, Oct. 25, pp. 15,368–9.

Nickel-Alloy-Steel Turbine Rotors

D. R. DEFOREST, D. L. NEWHOUSE and B. R. SEGUIN: 'Progress in the Development of Steam Turbine-Generator Rotor Materials.'

Amer. Soc. Mechanical Engineers, Paper 57-*A*-280, Dec. 1957; 19 pp.

Intensive investigations, in the laboratory and on a plant scale, have been carried out over the past few years with a view to determining factors which can contribute to failure of steam-turbine rotors, and methods of production control and inspection which can be used to ensure satisfactory service. Work on these lines in the U.S.A. has been co-ordinated through the A.S.T.M. Brittle Fracture Committee, the membership of which includes representatives from the principal manufacturers of large steam turbine generators and important producers of large rotor forgings.

The aim of this paper is to give the 'high lights' of results which have been obtained in a comprehensive Rotor Development Programme undertaken by the General Electric Company, Schenectady, N.Y., with the object of full-scale trial and comparison of rotor materials and manufacturing processes differing from those which have previously been used. The work covered tests on rotors typical of current supply (obtained from various makers in the U.S.A. and Europe) and study of the influence of composition and processing of steels normally used for this purpose and steels of modified composition. The types represented included nickel-molybdenum-vanadium, nickel-chromium-molybdenum, chromium-molybdenum, and nickel-chromium-molybdenum-vanadium. Full details are given of the modifications of compositions studied, and of variations in manufacturing procedure and in thermal treatment. Examination of the materials

comprised notched-disc spin tests, tensile tests, determination of Charpy impact-transition temperature, drop-weight tests, measurement of residual stress, and inspection for soundness, using conventional non-destructive methods.

Parallel with the evaluation, by these methods, of the possibility of achieving tougher and stronger forgings by major changes in material and processing practice, an effort was made to improve nickel-molybdenum-vanadium-steel forgings going into production units. The approach in this connexion was two-fold: lowering of the molybdenum content to about 0·30 per cent. (as compared with approximately 0·5 per cent. characteristic of this type of rotor steel) and modifications in thermal treatment (mainly lowered austenitizing temperatures). Statistics recorded in the paper demonstrate the improvement currently obtained by such methods: the changes have already resulted in consistent production of rotors in which the Charpy impact-transition temperature has been lowered from an average of 130°F. (54·5°C.) to 105°F. (40·5°C.) or below.

Although about half of the forgings covered by the Rotor Development Programme have still to be evaluated, the authors consider that results already available definitely indicate progress in ensuring consistently improved resistance to brittle fracture in the presence of severe stress concentration.

From the point of view of materials, the following opinions are expressed.

'For generator rotors having yield strengths of 75,000 p.s.i. or below (33·5 t.s.i.: 52·7 kg./mm.²) nickel-molybdenum-vanadium steel of controlled hardenability and lowered molybdenum content, melted by the basic-electric process and vacuum-poured to remove hydrogen, should provide optimum strength, ductility and toughness. For higher yield strengths, the necessary toughness, ductility and magnetic properties should be met by a nickel-chromium-molybdenum-vanadium steel similarly made, and containing about 0·22 per cent. carbon, 0·40 per cent. manganese, 2·0–2·8 per cent. nickel, 1·2–1·5 per cent. chromium and 0·5 per cent. molybdenum.

'For low-pressure high-yield-strength turbine rotors, the 3/0·5 chromium-molybdenum class of steel offers promise, in addition to the nickel-chromium-molybdenum-vanadium steels referred to above.'

Welding of Low-Alloy Steels: Recent Research in the U.K.

P. H. R. LANE: 'A Summary of the Work of the British Welding Research Association on Welding Low-Alloy Steels.'

Brit. Welding Jnl., 1958, vol. 5, Jan., pp. 38–42.

This article reviews investigations carried out under the auspices of The British Welding Research Association, following preliminary work by The Welding Research Council of The Institute of Welding. The ultimate aim of the research was the development of steels which would have satisfactory weldability combined with specified tensile properties. In 1945, at the stage at which The British Welding Research Association assumed responsibility, the terms of reference were as follows:

'To extend present knowledge of the influence of the various alloying elements on weld hardening and the tendency to crack of alloy steels when welded under specific welding conditions, with the ultimate object of recommending for commercial development steels having 0·2 per cent. of proof stresses, respectively of:

'(*a*) 25 tons/sq. in. (39·4 kg./mm.²),

'(*b*) 30 tons/sq. in. (47·3 kg./mm.²),

'(*c*) greater than 30 tons/sq. in. (47·3 kg./mm.²).'

The work comprised broadly the examination of the welding characteristics of several series of steels containing carbon of the order of 0·1–0·2 per cent. and varying combinations of manganese, nickel, chromium, molybdenum and vanadium. Concurrently with the study of the influence of composition on weldability and mechanical properties, an investigation was made of the mechanism of cracking in the weld-heat-affected zone, and consideration was given to tests for assessment of weldability. The scope of the work under the respective heads is summarized, together with the main conclusions drawn. An appendix to the report contains a description of some of the welding tests developed.

Most of the work described in this summary has been published in the form of reports or technical papers, and reference is made to it in the B.W.R.A. Publications entitled 'Arc Welding Low-Alloy Steels' and 'The Weldability of High-Tensile Structural Steels'. The major papers relating to the investigations have been abstracted in *The Nickel Bulletin* at the times of publication; see, for example, 1952, vol. 25, No. 2 p. 47; 1953, vol. 26, No. 7, p. 116; 1954, vol. 27, No. 10, p. 195; and 1956, vol. 29, Nos. 5 and 12, pp. 90 and 215.

Welding of High-Tensile Low-Alloy Steel, U.S.S. T-1

E. F. NIPPES, W. F. SAVAGE and R. J. ALLIO: 'Studies of the Weld Heat-Affected Zone of T-1 Steel.'

Welding Jnl., 1957, vol. 36, Dec., pp. 531s–40s.

The steel concerned in this investigation is one of several compositions which have recently been developed to give high yield strength, good weldability and satisfactory low-temperature toughness. The mechanical composition of the material used is given as carbon 0·15, silicon 0·23, manganese 1·00, sulphur 0·023, phosphorus 0·014, nickel 0·94, chromium 0·53, molybdenum 0·45, vanadium 0·05, copper 0·34, tin 0·008, titanium 0·004, boron 0·0014, per cent.

It is well recognized that in the case of hardenable steels, rapid cooling after welding is undesirable, since it results in formation of brittle martensite. The favourable mechanical properties of T-1 steel (of which details have been published by various investigators) are, however, contingent on the

existence of low-carbon martensite and since pre-heat or high energy inputs slow down the rate of cooling after welding (thus tending to produce microstructures other than low-carbon martensite), it could reasonably be presumed that both procedures would be detrimental to the notch toughness of the steel. It is known that in commercial practice pressure-vessel steels such as T-1 are often pre-heated for welding, and that if submerged-arc techniques are used, high energy inputs may be expected. It was therefore desirable to study the effect of weld thermal cycles involving slow cooling of continuous-cooling transformations, and the properties of welds made in the T-1 steel under various conditions.

The extensive work done on these three aspects is reported in detail in this paper. The work covered:

(*a*) measurement, by oscillographic techniques, of thermal cycles experienced in the heat-affected zone of T-1 steel welded under conditions of high energy input,

(*b*) determination of the continuous-cooling transformation behaviour, by means of thermal analysis and high-speed dilatometry,

(*c*) examination of the structure of the heat-affected zone of the steel under various welding conditions.

It is concluded that slow cooling rates have a damaging effect upon the notch toughness in the heat-affected zone and that this loss in toughness after slow cooling is caused by the presence of a microstructure consisting of martensite and bainite in a matrix of ferrite. A minimum cooling rate of 6·5°F./sec. (3·5°C./sec.) at 900°F. (482°C.) is required to produce satisfactory notch toughness as defined by the ability to absorb 10 ft.-lb. of energy at −50°F. (−45·5°C.). The weld energy input and initial plate temperatures producing this cooling rate were calculated for various thicknesses of T-1 steel.

Welded Austenitic/Ferritic Steel Joints

See abstract on p. 94.

HEAT- AND CORROSION-RESISTING MATERIALS

Vacuum Melting of Nickel-containing Materials

W. W. DYRKACZ: 'Vacuum Melting in the Steel Industry Today.'

Jnl. of Metals, 1957, vol. 9, Dec., pp. 1513-16.

It is anticipated that by the end of 1958 there will be available, in the U.S.A., facilities for the annual production of approximately 27,000,000 lbs. of materials by induction vacuum melting and of 100,000,000 lbs. by consumable-electrode vacuum arc melting. The increase in capacity over the 1957 figures represents, respectively, 7,000,000 and 50,000,000 lbs. This article gives details of the two techniques and discusses some of the advantages which have led to their increased commercial use.

Consideration is first given to induction vacuum melting, and the facilities available at the Watervliet works of Allegheny-Ludlum Steel Corporation are described. Alloys now being regularly melted by this method include the complex nickel-base alloys Waspaloy, M-252, Nimonic 90, Udimet 500, and R.235, and the cobalt-base alloy 1570. Induction vacuum melting is peculiarly suitable for these alloys, in that it makes possible control of titanium and aluminium contents within narrow limits. Moreover, in the vacuum-melted materials the elements are present in solid-solution form rather than as oxides, nitrides or carbo-nitrides, and there is thus a greater amount of effective titanium available for formation of the Ni_3TiAl precipitate which is the high-temperature strengthening agent.

The consumable-electrode vacuum-melting process involves re-melting, under controlled conditions, of air-melted electrodes of the desired composition. The main advantage of this technique, as compared with induction vacuum melting, is its ability to produce larger-size ingots and to minimize segregation. The method is applicable for production of high-temperature alloys, stainless steels, and low-alloy steels. Data presented in the paper illustrate the improvement in fatigue strength and room-temperature properties of a low-alloy steel, and the reduction in gas content of typical high-temperature alloys attainable by this method of melting. The process ensures high chemical homogeneity in alloys containing titanium and molybdenum, minimizes segregation of sulphur and non-metallic inclusions, and reduces gas content, giving an overall increase in purity and properties.

The article includes a *bibliography* of 32 items on vacuum melting.

Tensile Properties of Precipitation-Hardening Stainless Steels at Elevated Temperatures

I. M. KURG: 'Tensile Stress-Strain Properties of 17-7 P.H. and A.M. 350 Stainless-Steel Sheet at Elevated Temperatures.'

Nat. Advisory Committee for Aeronautics Tech. Note 4075, Sept. 1957; 16 pp.

This report is a further contribution to the study of the high-temperature properties of two materials which are considered to hold particular promise for use in the aircraft field.

Tensile stress-strain test results are presented for 17-7 P.H. sheet, in condition T.H. 1050 (see *Nickel Bulletin*, 1958, vol. 31, No. 1, p. 16), and for A.M. 350 in the double-aged condition (*Nickel Bulletin*, 1958, vol. 31, No. 1, p. 17). The range covered is room temperature to 1300°F. (704°C.). Stress-strain curves are shown, with tabular data on yield and ultimate tensile strengths, Young's modulus and elongation.

The tests were made on the same sample of material as that used for determination of the compressive properties reported in *N.A.C.A. Tech. Note* 4074 (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 1, p. 16). A comparison is made between the tensile and compressive characteristics of the materials.

The results demonstrate that 17-7 P.H. steel retains a large proportion of its room-temperature strength up to about 700°F. (370°C.), and that for A.M. 350 the corresponding temperature is about 800°F. (425°C.). At room temperature the ultimate tensile stress is about the same for the two materials, but the yield stress for A.M. 350 is appreciably less than for 17-7 P.H. Above 800°F. (425°C.), however, the strength of A.M. 350 is the greater. The compressive yield stresses for both of the steels are slightly greater than the corresponding tensile yield stresses, over the entire temperature range. The variation of Young's modulus with temperature is similar in the two steels, both in tension and in compression.

Cobalt-base High-Temperature Alloy: J-1570

R. W. GUARD and T. A. PRATER: 'Austenitic Alloy for High-Temperature Use.'

Trans. Amer. Soc. Metals, 1957, vol. 49, pp. 842-56; disc., pp. 856-61.

A large majority of austenitic alloys for high-temperature use derive their strength from the precipitation of carbides (usually those of molybdenum, tungsten and niobium) or from the compound Ni_3Al. It has been observed that titanium as Ni_3Ti is a relatively poor strengthening agent in nickel-base alloys, but preliminary evidence has been obtained that in cobalt-containing alloys of suitable composition it is possible to obtain satisfactory high-temperature properties in materials containing titanium without aluminium. With the advent of large-scale vacuum-melting facilities it has become commercially feasible to make alloys in which the titanium contents are sufficiently high to confer precipitation-hardening properties, without undue formation of nitrides and loss of titanium.

This paper describes a study of the effects of composition, treatment and processing variables, on a group of cobalt-nickel-chromium-base alloys containing molybdenum, tungsten and titanium. From preliminary tests a single optimum composition (shown below) was selected for intensive investigation of the effect of processing variables.

Composition of Alloy J-1570

Co %	Ni %	Cr %	W %	Ti %	C %	Fe %
39·0	29·0	19·9	6·6	4·1	0·20	1·3

(In view of the slightly greater strengthening effect of tungsten, it was decided to use it alone, rather than in conjunction with molybdenum.)

Evaluation was by creep-rupture tests at 1500°F. (815°C.) and 30,000 p.s.i. (13·4 t.s.i.; 21·1 kg/mm²).

Properties typical of those obtainable in this alloy after various forms of heat-treatment are given in the paper. The results demonstrated that there is an optimum ratio of solution elements (molybdenum and tungsten) to the precipitation element (titanium). It is found that the 1,000-hour rupture strength at 1600°F. (870°C.) is 15,500 p.s.i. (7 t.s.i.; 11 kg/mm²).

Ductile Chromium and Chromium-base Alloys

'Ductile Chromium and its Alloys.'

Proceedings of a Conference held under the auspices of the Office of Ordnance Research, U.S. Army, during the Metal Congress and Exposition of the American Society for Metals, 1955.

Published by *Amer. Soc. Metals*, 1957; 376 pp. Price: 6 dollars.

In the introductory chapter the significance of chromium is emphasized, and a condensed résumé is made of research which has been directed towards overcoming the brittleness which is the inherent limitation of the metal and of high-chromium alloys. The Conference was convened with a view to bringing together all the information, accumulated in various countries, which could be regarded as relevant to efforts to produce ductile chromium-base materials. The contributions were made by individuals responsible for research on the subject, by representatives of metal and alloy producers, and by some individuals interested in the purely engineering-user aspect. The scope of the papers contained in the five sections of the volume is indicated below:

I. Reviews of Research on Chromium in America, Europe and Australia.

II. Production of Chromium Metal by Various Methods.

III. Ductile Chromium Metal; Properties and the Influence of Impurities on Ductility.

IV. Effect of Gas on Chromium Metal.

V. High-Chromium Alloys.

The papers in Section V are briefly noted below.

(*a*) D. J. MAYKUTH and R. I. JAFFEE: 'Influence of Chromium Metal Purity on the Properties of Chromium Alloys', pp. 229-46.

A series of iodide-chromium-base binary alloys was prepared, containing 5-85 per cent. of the following high-purity elements: iron, nickel, cobalt, vanadium, tungsten, niobium, tantalum, molybdenum. Alloys which proved to be sufficiently ductile to be rolled to sheet were subjected, in strip form, to bend and tensile tests at room temperature. As-cast hardness of the alloys was also determined.

It was found that only alloys in the chromium-iron, -nickel, and -cobalt systems have a significant range of workable compositions. The limiting

chromium content for the alloys which can be rolled to sound sheet are listed below.

Limiting Chromium Content %		
Cr–Fe	Cr–Ni	Cr–Co
76	63	45

The chromium-rich chromium-nickel alloys show better combinations of strength and ductility than the chromium-iron and chromium-cobalt alloys tested.

Duplicate ingots of compositions in the workable range of the three groups of alloys were prepared from electrolytic chromium, from iodide chromium intentionally contaminated with oxygen, and from hydrogen-reduced electrolytic chromium. These ingots were processed by the same methods as those used for their pure iodide-chromium-base counterparts. It was found that oxygen, sulphur and nitrogen, in the amounts contained in commercial chromium, have a detrimental effect on chromium-rich chromium-iron and chromium-nickel alloys. These effects are most severe in an alloy containing chromium 61, iron 39, per cent., in which such contaminants caused both hot shortness and room-temperature brittleness. In chromium-iron alloys of lower chromium content and in the highest-chromium chromium-nickel alloys fabricated, the principal effect of the contaminants appears to be hot shortness.

(*b*) D. J. MAYKUTH and R. I. JAFFEE: 'The Mechanical Properties of Swaged Iodide-base Chromium and Chromium Alloys', pp. 247–54.

The paper reports a comparison of the properties of iodide chromium and of chromium-iron and chromium-nickel alloys, in the form of bar stock, with those previously reported for strip (*vide supra*). The tests also included determination of dynamic modulus of elasticity over the temperature range which is of interest in connexion with gas turbines.

The results showed that swaged chromium having a fibrous structure, tested in the form of round bar, has excellent room-temperature tensile ductility, but that this is lost after the material has been recrystallized. Swaged and recrystallized chromium-base alloys containing 50 per cent. of iron or nickel, tested as bar stock, have excellent tensile ductility, but ductility is low in an alloy containing 40 per cent. of iron and is lost completely in the 40 per cent. Ni chromium-nickel alloy.

The modulus of elasticity of chromium is 42,000,000 p.s.i. at room temperature, decreasing to 36,000,000 p.s.i. at 800°C.: a minimum occurs near room temperature. In the chromium-base alloys the modulus of elasticity increases at a chromium content of 50–60 per cent. but by only a relatively small amount.

(*c*) R. W. FOUNTAIN and J. L. LAMONT: 'Ductility and Toughness of High-Chromium Chromium-Iron Alloys', pp. 255–73; disc., pp. 273–6.

The influence of deoxidation practice and hot-working technique was studied as affecting the mechanical properties of chromium-iron alloys in the range 30–50 per cent. chromium. The effect of alloy additions, on the impact-transition temperature, was also investigated. Of the deoxidizers used, zirconium and beryllium were the most effective as final additions after initial deoxidation with carbon. Alloys treated with zirconium were relatively clean, generally of more uniform ductility, and of finer cast grain size. It was found possible, by tantalum and zirconium additions, to lower the impact-transition temperature of the 50–50 chromium-iron alloys from about 180°C. to 80°C. It is concluded that, by control of melting, deoxidation, and hot-working techniques, good ductility can be obtained in chromium-iron alloys up to at least 50 per cent. chromium.

(*d*) E. P. ABRAHAMSON and N. J. GRANT: 'The Chromium–Nickel–Nitrogen Ternary Diagram', pp. 277–86.

From the observations made, which are illustrated by graphs relating to various sections of the ternary diagram, the authors conclude that this system is likely to produce practically useful chromium-base alloys, since nitrogen additions as small as 0·25 per cent. shift the γ-nickel boundary up to 60 per cent. chromium. By suitable solution treatment, a face-centred-cubic structure can be retained in this alloy group and this structure should be associated with satisfactory workability. It is possible to disperse, into either lamellar plates or into nodules, the intergranular chromium phase which has given trouble in these alloys. The authors conclude that nitrogen additions above 1 per cent. will be unnecessary, since all the benefits derived from the shifting of the γ-nickel boundary are attained when this nitrogen content is reached, and high amounts would cause embrittling nitrides to form at the grain boundaries.

(*e*) E. P. ABRAHAMSON and N. J. GRANT: 'Some Structures and Properties of the Chromium–Nickel–Nitrogen Ternary System', pp. 287–304.

The study of the ternary system reported above was followed by investigation of the lamellar structure associated with the presence of nitrogen in the alloys. Data on structure, hardness, tensile and stress-rupture properties, and on workability, lead to the following conclusions:

In slowly cooled chromium–nickel alloys containing 40 to 80 per cent. of chromium, addition of nitrogen causes precipitation of α-chromium in a lamellar form, as non-thickening discs. Rate of precipitation is dependent on temperature and on the chromium and nitrogen contents. The structure can be formed also by severely cold-working a solution-treated γ-nickel structure, followed by ageing. Hardness of the ternary alloys is dependent on solution temperature and on composition, and these factors also

affect the room-temperature ductility of the alloys. Ultimate tensile strength increases with increasing chromium and nitrogen contents. Stress-rupture properties are low in comparison with those of commercial alloys, but ductilities are comparable. Chromium-nickel-nitrogen alloys containing up to 50 per cent. of chromium can be severely cold-worked, and workability is improved, if the alloys are first solution-treated to retain the γ-nickel structure.

(*f*) H. KATO and E. T. HAYES: 'Mechanical Properties and Toughness of Chromium-Iron Alloys', pp. 305–13.

This report covers a study of the influence of oxygen (20 to 2,500 p.p.m.) in chromium-iron alloys. Particular attention was devoted to the 50–50 composition, but some work was done also on alloys containing 25–60 per cent. of chromium.

Experience in fabrication, supplemented by tensile, hardness and impact tests, together with creep-rupture tests at 815°C. (reported from another source) showed the following effects:

Room-temperature toughness is apparently unaffected by oxygen within the limits studied, but brittle-to-ductile transition temperature is lowered by reduction of oxygen content. Alloys containing the higher amounts of oxygen had greater tensile strength than those of lower oxygen content, but there was little difference in elongation values among the alloys examined. Increasing the chromium content of the alloys resulted in higher tensile strength and greater hardness, but impaired ductility. The creep-rupture properties were also improved by increase in chromium content.

(*g*) D. P. MOON, H. A. BLANK and A. M. HALL: 'A Forgeable Chromium-Iron Alloy', pp. 314–21.

The general objective of the work described was development of a forgeable chromium-rich chromium-iron alloy, and, more specifically, investigation of a composition of chromium 60, iron 25, per cent. with not more than 2 per cent. of critically scarce elements. The specification required that the material should have stress-rupture properties equal to those of forged S-816,* and room-temperature ductility adequate to ensure safe handling. It was also required that the alloy should be producible from commercially available materials, using conventional melting practice.

The work led to development of alloys of the composition chromium 70, iron 30, molybdenum 9, titanium 2–3, aluminium up to 0·5 (parts by weight). Such alloys are malleable at room temperature, but do not show measurable ductility in room-temperature tensile tests. They have excellent resistance to thermal shock.

* Complex cobalt-base alloy containing about 20 per cent. each of nickel and chromium, with smaller percentages of molybdenum, tungsten and niobium.

(*h*) P. DUWEZ and H. MARTENS: 'The Kinetics of the Formation of the Sigma Phase in Chromium-Iron Alloys', pp. 322–35.

Chromium-base alloys containing about 40–50 per cent. chromium, although characterized by good properties at high temperatures, have only a limited field of application, due to inherent brittleness. One reason for such brittleness is the presence of the sigma phase, and the study reported in this paper was undertaken to establish the fundamental rules governing the rate of decomposition of alpha solid solution into sigma. Chromium-iron alloys and ternary alloys of chromium and iron containing also 3 per cent. of tungsten were used.

In binary chromium-iron alloys sigma forms by a process of nucleation and growth. The rate of reaction, which is at a maximum at about 44·7 per cent. chromium, is a function of temperature, increasing with rising temperature up to 725°C. and then decreasing, becoming very slow as the temperature approaches the equilibrium between alpha and sigma. Cold working greatly accelerates formation of sigma, but does not appreciably change the temperature at which the rate is maximum. Addition of 3 per cent. of tungsten was found to affect the rate of sigma formation and to raise the equilibrium temperature. The amount of nitrogen present in the ternary alloys also affects the rate of sigma formation.

(*i*) A. K. WONG: 'Short-Time Elevated-Temperature Tensile Properties and Notch Toughness of Some Chromium-Iron Alloys', pp. 336–42.

Short-time tensile tests were made, at elevated temperatures, on 50–50 and 40–60 chromium-iron alloys which had undergone identical degrees of hot working and similar heat-treatment. Strength decreased as the temperature of test rose, up to 500°F. (260°C.), and subsequently increased with further rise in temperature, reaching a maximum at 1000°F. (540°C.), with corresponding decrease in ductility. In general, the strength of the 50–50 alloy was higher than that of the 40 per cent. chromium alloy, but the notch toughness of the lower-chromium alloy was the better of the two.

(*j*) W. D. FORGENG and G. D. MOTOCK: 'Metallography of Electrolytic Chromium', pp. 343–61.

The microstructure of chromium metal can be satisfactorily revealed by electrolytic etching with a dilute chromic/sulphuric-acid solution. The microstructure of as-deposited chromium shows local variations which are apparently the result of momentary changes in the plating conditions. The most common structure in the as-deposited metal is a difficultly-etching hard white phase showing crack patterns. Only slight change takes place in the microstructure or microhardness on heating in vacuum for 30 minutes at temperatures up to 500°C., but treatment at higher temperatures results in recrystallization and grain growth, and the metal

becomes progressively softer. Electron-microscopic examination reveals the presence of minute trans-crystalline cracks. Treatment of electrodeposited chromium with hydrogen at 500°C. produces a metal which is somewhat softer than iodide chromium, and this treatment also reduces the oxygen, hydrogen, nitrogen, carbon and sulphur contents to very low values.

Iron-Nickel-Chromium-Nitrogen System: Constitution

G. F. TISINAI and C. H. SAMANS: 'Phase Relationships in the Fe-Cr-Ni-N System.'

Trans. Amer. Soc. Metals, 1957, vol. 51, *Preprint* 71; 16 pp. — tables and figures.

During recent years considerable interest has arisen in the austenitizing power of nitrogen in iron-chromium-nickel alloys, in which it is used in partial substitution for nickel. The authors begin their paper with a succinct but comprehensive review of the literature, which reveals that most of the work done in this connexion has been concerned with specific compositions, and that there has been little study of the system as a whole. There was therefore no reliable concept of the phase fields existing in the quaternary alloys. The investigation reported in the present paper had the three-fold aim of determining the austenite/austenite+ferrite boundaries in the system, examination of compositions higher in chromium and lower in nickel than those which had been previously studied, and investigation of the physical metallurgy of the alloys at temperatures between 1300° and 2200°F. (705° and 1205°C.).

Some 80 alloys were used, giving compositions in the range chromium 21–23, nickel up to 20, nitrogen up to 1, per cent., balance iron. About 60 were prepared by powder-metallurgy methods.

The main conclusions drawn from the extensive data reported are summarized below.

At 2200°F. (1205°C.) the austenite/austenite+ferrite boundary is a flat plane running, for nickel-free alloys, from about 0·40 per cent. nitrogen at the 21 per cent. chromium level, through about 0·80 per cent. nitrogen at the 27 per cent. chromium level, and through about 1·15 per cent. nitrogen at the 33 per cent. chromium level. At all these chromium levels nickel can be substituted for nitrogen in the ratio of 1 per cent. nickel for each 0·025 per cent. nitrogen.

At 2000°F. (1093°C.) there is a clear-cut separation between the austenite and the austenite+ferrite fields only at the 21 per cent. and 24 per cent. chromium levels. With higher chromium contents, there is a tendency to formation of nitride precipitates unless the nickel content is high.

It appears that for each composition there is an optimum austenitizing temperature, i.e., a solution-annealing temperature at which the maximum amount of austenite is produced. Treatment at temperatures above the optimum tends to cause formation of delta ferrite, and the use of temperatures lower than the optimum causes formation of nitrides and thus tends to increase ferrite content, as a result of an impoverishment in nitrogen.

Below the optimum austenitizing temperatures the microstructures of solution-treated iron-chromium-alloys containing nickel and nitrogen change considerably as a result of nitride precipitation: depending on the degree of precipitation and the original composition, considerable amounts of martensite or ferrite may form. Since, however, this precipitation does not result in the matrices of the alloys becoming significantly depleted in nickel; they retain some austenite in their structure even after extensive precipitation of nitrides (and carbides). This is in direct contrast to effects in similar alloys which contain only carbon and nitrogen, where the matrix decomposes completely to ferrite.

Sigma phase was found, after nitride precipitation, only in the alloys at the 33 per cent. chromium level, indicating that in lower-chromium alloys nitride (and carbide) precipitation reduced the effective chromium content in the matrix to a level at which sigma formation occurred only very slowly, if at all.

Ageing of Iron-Nickel-Chromium-base Alloys Hardened with Titanium

T. W. EICHELBERGER: 'The Response of an Iron-base Alloy, Hardened with Titanium, to Various Ageing Times and Temperatures.'

Trans. Amer. Soc. Metals, 1957, vol. 51, *Preprint* 60; 29 pp.

The investigation reported was planned to determine the influence of ageing conditions on the mechanical properties of some high-temperature alloys in which titanium is the precipitation-hardening medium. The work may be considered in association with that which led to the development of the 'genuage' cycle of precipitation hardening (see *Trans. Amer. Soc. Metals*, 1957, vol. 50, *Preprint* 21; *Nickel Bulletin*, 1958, vol. 31, No. 2, pp. 56–7.)

The research now reported was concerned with the influence of composition, ageing temperature and ageing time, on the hardness attainable in alloys within a composition range typical of three commercial materials: Tinidur, Discaloy and A-286 (chromium 13–16, nickel 20–30, carbon 0·1 max., manganese up to 2, silicon up to 1, titanium 1·3–2·5, vanadium 0·25–0·5, per cent.). Particular attention was given to the influence of soluble and insoluble titanium.

The specimens were solution treated for 1 hour at 1950°F. (1065°C.) and the temperatures of the ageing treatments were 1200°, 1300°, 1350° and 1400°F. (650°, 705°, 735° and 760°C.). In addition, the influence of time of ageing was studied, on specimens aged (after the same solution treatment) for various periods at 1100°, 1500° and 1600°F. (595°, 815° and 870°C.). Although no extensive study was made of the influence of the solution temperature on the properties attained in the subsequent ageing, some specimens were solution treated at 1800°F. (980°C.) for 1 hour and aged at 1350°F. (735°C.), for comparison with others which had been solution treated for 1 hour at 1950°F. (1065°C.) and aged at

1350°F. (735°C.). This variation in solution temperature caused no change in response to the ageing treatments.

Master curves, derived from the results, evaluate (1) hardness as a function of time, temperature and composition; (2) maximum hardness attainable as a function of temperature of ageing and composition of the alloys, and (3) time to reach maximum hardness, as a function of ageing temperature.

The main conclusions reached are given below:

The alloy system investigated shows age-hardening at temperatures between 1100° and 1600°F. (593° and 870°C.).

Soluble-titanium contents of 1·3 per cent. and below did not produce sufficient hardening to be of much practical importance.

Molybdenum above 2¼ per cent. produces hardening above 1400°F. (760°C.). Vanadium does not visibly affect the ageing reaction.

Time to attain maximum hardening, as a function of ageing temperature, gives an activation energy, for the hardening reaction, of 72,500 cal/mole between 1200° and 1400°F. (648° and 760°C.).

At temperatures of 1200°–1400°F. (648°–760°C.) 95 per cent. of the precipitate is nucleated in the first 8 hours of the ageing reaction.

Precipitation Phenomena in Nickel-Aluminium Alloys

J. MANENC: 'Precipitation of the Ni_3Al Phase in an Alloy containing 7·8% of Aluminium.'

Revue de Métallurgie, 1957, vol. 54, Nov., pp. 867–78.

In 1955 the author presented a report on X-ray study of the mode of precipitation occurring in a nickel-chromium-base alloy of the 80–20 type hardened with aluminium and titanium (*ibid.*, 1957, vol. 54, pp. 161–8). The results confirmed conclusions drawn from investigations made by other techniques, showing that, prior to visible precipitation of the hardening constituent, there is perceptible disturbance of the solid-solution matrix.

This later paper records extensive observations of a similar nature made on a binary nickel-base alloy containing 7·8 per cent. of aluminium, and the conclusions are supported by results of less detailed examination of three other related nickel-base alloys containing, respectively, 6·8 per cent. aluminium; 2 per cent. titanium+5 per cent. aluminium; 7 per cent. titanium+ 2 per cent. aluminium.

The results further confirm the 'pre-precipitation disturbance' which occurs locally in the solid-solution matrix when the alloys are isothermally treated for suitable periods at temperatures within the range 500°–800°C. The precipitating phase is Ni_3Al. Typical structures are illustrated in the paper.

Oxidation of Molybdenum

E. S. JONES, J. F. MOSHER, R. SPEISER and J. W. SPRETNAK: 'Oxidation of Molybdenum.'

Corrosion, 1958, vol. 14, Jan., pp. 2t–8t.

The authors introduce their report by a useful review of relevant literature: the *bibliography* supporting the paper numbers 29 items. Particular attention is given to the nature and effect of the 'catastrophic oxidation' which is at present severely restricting the use of molybdenum-containing materials at elevated temperatures.

The extensive research reported in this paper covered oxidation of molybdenum (in the form of sheet and wire) heated in still air and in metered moving air, at temperatures in the range 900°–1800°F. (482°–983°C.). Full details are given of the apparatus used in the experiments, methods of preparing specimens, and test results. The information given includes data on rates of sublimation of MoO_3 at various temperatures, condition of the oxidized specimens and the nature of the oxides formed. The mechanism of formation of the oxides is also discussed.

Resistance of Aluminium-Coated Steel to Corrosion by Fuel Ash

J. E. SRAWLEY: 'Fuel-Ash Attack on Aluminum-Coated Stainless Steel.'

Corrosion, 1958, vol. 14, Jan., pp. 36t–8t.

This is a condensed version of *U.S. Naval Research Laboratory Report N.R.L.* 4838, Oct. 1956; see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 7–8, p. 140.

Under the conditions of test used, aluminium-coated 25–20 chromium-nickel stainless steel was not protected from fuel-ash corrosion by hot-dip coating with aluminium.

Structural Sandwich Design and Materials

See abstract on p. 78.

Nickel-Alloy Springs

'High-Nickel Alloy Spring Materials.'

Electrical Manufacturing, 1957, vol. 60, Sept., pp. 102–7.

Springs incorporated in electrical and electronic components are often required not only to operate under heavy static and cyclic loads, but also to exhibit satisfactory resistance to elevated temperatures and severe corrosive attack. While several materials will meet one of these requirements, comparatively few will function satisfactorily when subjected to a combination of such conditions. A number of high-nickel alloys are, however, outstandingly useful in such cases. In this article the suitability of nickel alloys as spring materials is reviewed on the basis of information supplied by The International Nickel Company, Inc. Discussion centres on Monel (which can be regarded as a general-purpose alloy), the high-temperature alloys Inconel and Inconel X, and the age-hardenable types, K Monel, Duranickel and Permanickel. These alloys are evaluated in sections dealing with resistance to elevated temperatures, corrosion-resistance, electrical conductivity and magnetic properties, and design factors. Information given in the text is supplemented by comprehensive tabular and graphical data.

Particular attention is drawn to the importance of

load-carrying capacity, or relaxation properties, of spring materials, as influenced by static elevated temperature or fluctuating-temperature conditions. Data on the comparative relaxation properties of various materials, in terms of temperature and applied stress, demonstrate the useful qualities of the nickel alloys and indicate the limiting temperatures up to which the respective materials may usefully be employed. In connexion with temperature effects, mention is made also of the increase in strength and ductility which occurs in most nickel alloys at sub-zero temperatures.

Resistance to corrosion is a further factor of considerable importance in spring alloys, in particular resistance to pitting attack. Pits act as stress raisers which, in wire of fine diameter operating under high or cyclic stress, can cause premature failure. Monel, stainless steel, Inconel, Inconel X and the other high-nickel alloys are all characterized by good resistance to corrosion, but it is recommended that empirical tests should be made before use of the individual alloys under specific corrosive conditions.

For springs in which high electrical conductivity is required, the use of Permanickel is recommended: typical physical and mechanical properties of this alloy are listed. Reference is made also to conditions in which spring materials having non-magnetic properties are required: in such cases K Monel, Inconel, Inconel X and, in certain conditions, Duranickel, are useful.

Data on permissible design stresses at various temperatures show that at room temperature the endurance limit and fatigue strength of high-nickel alloys are lower than those of spring steels of identical tensional and torsional moduli, but that at elevated temperatures and in corrosive atmospheres the nickel alloys are much superior. Shot peening is not, in general, recommended for nickel-alloy springs, since minute particles from the steel shot may become embedded in the surface of the wire and serve as focal points for concentration-cell corrosion.

The review closes with brief notes on five types of heat-treatment used in processing some or all of the alloys.

Stainless Steel for Nuclear-Power Plants: Cobalt Content

J. R. LANE: 'Is Cobalt Harmful in Stainless Steel ?' *Metal Progress*, 1957, vol. 72, Dec., pp. 86–7.

Cobalt can occur, in residual amounts, as an impurity in some types of nickel used in stainless steels. In most applications for which such materials are used, cobalt of that order does not affect the service behaviour, but for certain uses in nuclear reactors even small amounts of cobalt are undesirable, because, when subjected to neutron bombardment in a reactor, an atom of natural cobalt can absorb a neutron and be thereby converted to a new isotope, cobalt-60. This isotope is unstable, and decays, with a half-life of 5·2 years, by emission of gamma rays. When a reactor is shut down the activated cobalt continues to give off its radiation, which is both intense and long-lived.

The problem is being solved by the use of special grades of highly-purified nickel. Specifications have been drawn up for two grades of nickel-chromium steel for reactors: one (A.I.S.I. Type 348) is low in tantalum (a neutron absorber); the second (Type 349*) is also low in tantalum and in cobalt. The compositions of the two grades and of the parent Type A.I.S.I. 347, from which they are derived, are shown in the table below.

Resistance of Nickel-containing Aluminium Alloys to High-Temperature Water

K. M. CARLSEN: 'Structural Features of Corrosion of Aluminum Alloys in Water at 300°C.' *Corrosion*, 1958, vol. 14, Jan., pp. 53t–5t; disc., pp. 55t–6t.

Draley and Ruther have shown that aluminium alloys containing cathodic elements have good resistance to corrosion by water at elevated temperatures. Such alloys, although showing improved resistance to sudden breakdown, have fairly high all-over corrosion rates, and the study reported in this paper was initiated to provide information on the factors determining this rate of corrosion.

The following types of aluminium-base alloy were used in the tests: aluminium-nickel, -iron, -nickel-iron, -copper, and -nickel-silicon. The corrosive medium was distilled water at 300°C. Photomicrographs show typical structures of the alloys after exposure.

The observations made by the author lead him to conclusions which vary in some respects from those reached by Draley and his co-workers, with reference to the nature of the corrosion attack and of the protective effect resulting from alloying.

Comments by DRALEY and RUTHER, and CARLSEN'S reply, follow the paper.

Type	C %	Mn %	Si %	Cr %	Ni %	Nb+Ta %	Ta %	Co %
347	0·08 max.	2·00 max.	1·00 max.	17-19	9-13	10xC min.	—	—
348					9-13		0·10 max.	—
349*					9-12		10% max. of Nb content	0·20 max.

* Not yet standardized by The American Iron and Steel Institute.

Resistance of Metals to Corrosion by Cement-Water Mixture

D. MATOUSCHEK: 'Corrosion of Metals by Cement Slurries.'

Zement-Kalk-Gips, 1957, vol. 10, Apr., pp. 124–7.

The paper presents the results of an investigation of the relative resistance to corrosion, by a cement slurry (one part cement to four parts water), of the following metals and alloys: a nickel-chromium alloy (of unspecified composition), iron, tin, zinc, copper, brass, magnesium, aluminium, four aluminium alloys, tin solder, and lead. Specimens were half-immersed in the slurry for a period of 360 days. The plates were periodically cleaned and weighed, and corrosion rates were calculated from loss or gain in weight. The nickel-chromium alloy was found to be the most resistant to attack: iron and tin also showed satisfactory resistance. Zinc, copper and brass were reasonably resistant, but magnesium, aluminium and the aluminium alloys, tin solder, and lead were severely corroded and are considered unsuitable for applications involving contact with cement.

Corrosion in Catalytic Reformers

G. SORELL: 'Compilation and Correlation of High-Temperature Catalytic Reformer Data.'

Corrosion, 1958, vol. 14, Jan., pp. 15t–26t.
Contribution to studies by N.A.C.E. Technical Group Committee T.8.

The recent phenomenal growth of catalytic reforming, into a major oil-refining process for production of high-octane motor fuels, has caused widespread concern on account of serious corrosion problems encountered in this type of process. A recent survey report presented to the American Petroleum Institute stated that the majority of 31 companies interviewed had experienced excessive metal loss, associated with serious operational difficulties, the most common of which involved plugging of process equipment by loose corrosion products consisting predominantly of metal sulphide. The corrosive agent responsible was identified conclusively as hydrogen sulphide in the hot hydrogen-rich gas mixtures commonly encountered in catalytic reforming units.

Some of the principal petroleum-refining companies have initiated corrosion investigations, and several useful papers resulting from such studies have recently been published: the author directs attention to individual examples of such reports. In an effort to explore this problem further, two industrial 'study committees' were formed (the A.P.I. Panel on Reformer Corrosion and the N.A.C.E. Task Group T-5B-2). The participating membership of both committees is drawn from practically all the major American petroleum-refining companies. Pooling of knowledge and experience among the supporting members has already made an important contribution to the better understanding of the problems involved and the primary object of the present paper is to correlate, in convenient reference form, relevant high-temperature corrosion data which have thus become available and to present the findings in a form useful in connexion with design of process equipment. With this aim, measurements of corrosion rate obtained from published literature and from hitherto unpublished reports are presented in tables and graphs: the data originate from a wide range of sources, covering laboratory investigations and operating experience. The total number of individual measurements embodied in the summary is estimated to be well over 1,000. [The field corrosion data in this report relate exclusively to catalytic reforming plants and are not concerned with the more corrosive conditions typical of catalytic desulphurization units. A paper summarizing information on the latter aspect will be compiled at a later date when more service experience on such process units becomes available.]

The author's discussion of the statistics and other information presented is restricted to a review of factors considered to be practically useful to the engineer in interpreting and evaluating the data and in selecting materials of construction for use in reforming units. Some extracts from the various sections of the review are given below.

Alloy Content of Steel

The accumulated data show, almost without exception, that chromium-molybdenum steels (up to about 9 per cent. chromium) are not superior to plain carbon steel, and there is some evidence that a 5 per cent. chromium ½ per cent. molybdenum composition may even be inferior in corrosion-resistance to unalloyed steel. Intermediate straight-chromium steels (11–16 per cent. chromium) show only a moderate degree of improvement over carbon or lower-alloy types. The 18–8 chromium-nickel grades all possess equally good corrosion-resistance, showing rates of attack only about one-fifth to one-tenth of those suffered by carbon and lower-alloy steels. The more highly alloyed austenitic steels (25–12 and 25–20 chromium-nickel types) are equal or superior to the 18–8 grades.

Environmental Factors Influencing Corrosion

Two major variables controlling the extent of corrosion at elevated temperatures in catalytic reformer units are temperature and the hydrogen-sulphide content of the gas mixtures handled. Other variables which influence rate of corrosion in such environments are pressure, hydrogen content, length of exposure and cyclic conditions. Corrosion rate approximately doubles for each 100°F. (56°C.) rise in temperature: pressure may considerably affect rate of corrosion, but the influence of hydrogen content is much less serious. Accumulated evidence from laboratory studies indicates that initial corrosion rates are very high, but that they fall off rapidly with increasing length of exposure. Laboratory data on the effect of time are, however, of only limited value to the designer and operator of large-scale equipment, since it is not possible in the laboratory fully to evaluate the rôle of the

sulphide corrosion scale in stifling further corrosion. A realistic appraisal of the time effect can be gathered only from experience in operating equipment. Cyclic process conditions have an accelerating effect on corrosion, but quantitative evaluation is difficult.

The author critically discusses the accuracy of the laboratory and large-scale data presented, with notes on possible sources of error, scatter of results, etc. It is considered that, in spite of certain discrepancies in the results reported, there is overall good consistency.

General Review of Corrosion Occurring in Catalytic Reformer Units and of Materials Used

It is pointed out that elevated-temperature corrosion by hydrogen sulphide, as experienced in catalytic-reforming and catalytic-desulphurizing units, differs in two important respects from the well-known sulphide attack commonly encountered in other types of refinery process where the sulphur is predominantly in the form of organic compounds. Firstly, hydrogen sulphide is more corrosive than sulphur compounds such as mono- and di-sulphides, mercaptans and thiophenes. Secondly, there is the distinguishing characteristic of the lack of resistance to corrosion by hydrogen sulphide offered by the chromium-molybdenum steels, which is in striking contrast to their behaviour under all other forms of sulphide attack in conventional types of refinery unit processing sour stocks.

The sulphide-scale formations commonly found in the high-temperature portions of catalytic reforming units are bulky, brittle and porous and have a general tendency to spall off. These physical characteristics usually render the scale ineffectual as a barrier to further corrosion. A further operational difficulty associated with scaling is the insulating effect in furnace tubes, which may lead to local overheating and subsequent rupture.

The author also draws attention to certain other forms of corrosion which may occur in reformer units: high-temperature hydrogen attack and lower-temperature galvanic or electrochemical corrosion. A form of normal-temperature attack occasionally found in catalytic reforming equipment is the failure, by cracking, of brass and austenitic steels. There is evidence that such failure occurs only under idle conditions, but it has not yet been established whether they are the result of stress-corrosion cracking, intergranular attack, or a combination of the two.

Solutions to the Corrosion Problems

As in most corrosion problems, counter-measures possible in reformer units involve either changing the environment or employing more resistant materials of construction. Some improvement can be obtained by scrubbing the re-cycle gas in a re-circulating amine-absorption system or by desulphurizing the feed stock in pre-treatment equipment, using a cobalt-molybdenum catalyst. Amine treatment is, however, only partially effective, since, while preventing build-up of hydrogen sulphide in the re-cycle gas, it does not protect the reaction train from corrosion by the hydrogen sulphide produced during passage of the feed naphtha.

The other method of overcoming corrosion is by the use of more suitable materials of construction. In this connexion, the notes on the corrosion-resisting properties of the respective types of steel given above indicate the outstanding suitability of the austenitic stainless steels. Some work has also been done on the protection of the lower-alloy steels by aluminium surface coatings. A considerable measure of success has been obtained along these lines.

Hydrogen-Sulphide Corrosion of Stainless Steels

E. B. BACKENSTO, R. E. DREW, J. E. PRIOR and J. W. SJOBERG : 'High - Temperature Hydrogen - Sulphide Corrosion of Stainless Steels.'
Corrosion, 1958, vol. 14, Jan., pp. 27t-31t.
Report prepared for N.A.C.E. Technical Group Committee T-8.

The subject matter of this report is closely related to that of the paper referred to in the preceding abstract. It deals with a particular aspect of the problem of hydrogen-sulphide corrosion encountered in catalytic reformer units, which is liable to interfere seriously with smooth and continuous operation. Data recorded in the paper by SORELL (*vide supra*), and evidence from various other sources, confirm that only the austenitic stainless steels offer satisfactory resistance to high-temperature hydrogen-sulphide attack under a wide range of conditions.

In order to utilize these materials most economically, however, it is necessary to recognize that in some conditions even the austenitic steels corrode at a rate sufficiently rapid to cause extensive formation of scale and consequent interference with plant operation. Tests have been made by Socony Mobil Oil Company to define more closely the behaviour of various types of high-chromium, austenitic chromium-nickel and lower-alloy steels in conditions involving hydrogen - sulphide attack at elevated temperatures. Following a review of background information from the earlier literature, a report is made of extensive laboratory tests on four types of high-chromium steel, in comparison with a 0·5 per cent. chromium steel and a typical grade of austenitic chromium-nickel steel. The tests covered exposure at temperatures ranging from 500° to 1400°F. (260° to 760°C.) and hydrogen-sulphide concentrations varying from 0·03 to 10 per cent. by volume. The data resulting from these tests are supplemented by reports of behaviour in a commercial catalytic reformer unit in which steels containing 0·9 or 12-16 per cent. of chromium and a high-alloy chromium-nickel steel were tested. Among the conclusions to be drawn from these tests are the following:

(i) For temperatures in the operating range of most catalytic reformers and naphtha desulphurizers the austenitic stainless steels are the most resistant to hydrogen-sulphide corrosion and the carbon and low-chromium steels are least resistant.

(ii) The corrosion rates of steels containing 0-5 per cent. chromium increase with rise of temperature up to about 1000°F. (540°C.). Beyond this temperature rate of corrosion slows down. Corrosion rates of the stainless steels also increase with rising temperature and the increase continues up to 1400°F. (760°C.), the highest temperature for which the authors obtained data. At some temperature between 1000° and 1400°F. (540° and 760°C.) the corrosion rates of the stainless steels become greater than those of the 0-5 per cent. chromium steels. It should, however, be noted that at 1400°F. (760°C.) none of these steels has sufficient resistance to hydrogen sulphide to be useful at any except very low concentrations.

(iii) The laboratory tests showed that at temperatures below 1000°F. (540°C.) rates of corrosion of the 12 per cent. chromium steels are not much slower than those found for the lower-chromium series, except at low concentrations of hydrogen sulphide. Within the range up to about 1000°F. (540°C.) the behaviour of the straight-chromium steels in relation to composition is somewhat irregular. For temperatures below 1000°F. the corrosion rates for the austenitic stainless steels are less than one-tenth of those found for the 0-5 per cent. chromium types.

Having thus confirmed the overall suitability of the austenitic steels for such service, the authors carried out tests to determine the influence of composition and processing variables on the resistance of the steels to hydrogen-sulphide attack.

Steels of normal 18-8 type, extra-low-carbon 18-8, niobium-stabilized 18-8 and 25-20 chromium-nickel types were subjected to two 'sensitizing' treatments (representative of heating conditions which might occur during welding) and to a stabilizing treatment (typical of conditions used to obviate intergranular corrosion). In almost all cases the heat-treatment caused a significant reduction in corrosion rate of the wrought specimens and the same effect was observed for a weld metal tested.

A second series of tests comprised exposure to hydrogen-sulphide attack, at 750° and 975°F. (400° and 525°C.), of typical cast austenitic steels and representative weld metals, in comparison with wrought steels. Both the weld metals and the cast steels showed lower corrosion rates than those characteristic of the wrought metals.

Tests on manganese-modified austenitic steels indicated that below 900°F. (480°C.) corrosion rates of such materials would be of the same order as for the conventional austenitic chromium-nickel steels. Above that temperature high-manganese steels may undergo somewhat greater corrosion than the chromium-nickel types.

Tests were made also on some high-alloy nickel-chromium-iron materials which are of particular interest on account of their proved resistance to stress-corrosion cracking. The types selected, Incoloy (nickel 34, chromium 21, per cent.), Inco 804 (nickel 42, chromium 30, per cent.) and Inconel (nickel 78, chromium 15, per cent.), were tested at 750°F. (400°C.) with a hydrogen-sulphide concentration of 1·5 per cent. by volume. The alloys showed broadly the same degree of resistance as has been found for the austenitic stainless steels.

Some preliminary tests were made to determine the possible cause of stress cracking, which has from time to time occurred in austenitic steels in reformers. The authors know of no case of such stress-corrosion having occurred during actual operation of a commercial unit, but from time to time failures of this type have been observed during rest periods and it is believed that the cracking may be due to the action of polythionic acids formed by the action of moisture on iron sulphide. Laboratory tests made in this connexion are described, with illustration of the specimens and general set-up used. The conditions involved exposure of stressed specimens to a high-temperature sulphide atmosphere sufficiently long to build up a scale, and subsequent exposure to a condensing-steam atmosphere. The results of these preliminary tests are considered to be inconclusive, and the authors urge that further work is required to determine fully the rôle of polythionic acids in causing stress-corrosion of austenitic stainless steels.

Corrosion by Fuming Nitric Acid: Inhibition by Hydrofluoric Acid

D. M. MASON, L. L. TAYLOR and J. B. RITTENHOUSE: 'Inhibiting Effect of Hydrofluoric Acid in Fuming Nitric Acid, on Liquid and Gas-Phase Corrosion of Several Metals.'

Corrosion, 1957, vol. 13, Dec., pp. 821t-7t.

In earlier experiments made to determine the suitability of various metals and alloys for use in contact with fuming nitric acid (as used in rocket propulsion) it had been shown that the presence of 0·4-0·6 per cent. of hydrofluoric acid was effective in inhibiting corrosion (*ibid.*, 1957, vol. 13, pp. 321t-8t; *Nickel Bulletin*, 1957, vol. 30, No. 7-8, p. 141). In those tests the specimen surfaces were exposed simultaneously to both liquid and gaseous phases: this later paper reports subsequent tests, the aim of which was to study *separately* the inhibiting influence of hydrofluoric acid on corrosion by (*a*) the liquid phase, and (*b*) the gas phase of fuming nitric acid. The scope of the investigation was extended to evaluate the corrosion-resistance of welded and unwelded aluminium alloys; chromium; A nickel; lead; tantalum; titanium alloys; carbon steel; chromium low-alloy and stainless steels, and chromium-nickel stainless steels of normal, free-cutting, precipitation-hardening and stabilized types.

The specimens were exposed, in apparatus which is described and illustrated, to the liquid or gas phase of fuming nitric acid containing 11-13 wt. per cent. NO_2 and 2-4 wt. per cent. H_2O, with or without addition of 0·4-0·6 per cent. of hydrofluoric acid. The main series of the tests was carried out at 130°F. (55°C.), and, in addition, the effect of fluctuations in temperature, such as might be

expected to occur in outdoor storage, was determined (on the niobium-stabilized 18-8 steel and an aluminium alloy) by cycling the temperature between 70° and 160°F. (20° and 70°C.) once every 24 hours. These two materials, and carbon steel, were also exposed, at 130°F. (55°C.), to nitrogen dioxide in the liquid and gas phases.

Results, where comparison was possible, confirmed those reported in previous investigations. In general, the presence of hydrofluoric acid inhibited corrosion of the aluminium alloys in both the gas and liquid phases of fuming nitric acid. These materials are recommended for use with inhibited acid, and are found to be fairly stable in the presence of uninhibited acid. Similar conclusions were reached in the case of 18-8 types of chromium-nickel steel, which were passivated in both phases of the inhibited acid if they were freshly exposed without destruction of the natural oxide film. If, however, previous extensive corrosive attack has occurred on any areas of the stainless steels, the presence of hydrofluoric acid in the fuming nitric acid aggravates the corrosive attack at these anodic areas.

In straight-chromium steels inhibition by hydrofluoric acid in liquid fuming nitric acid was achieved only if the steels had been previously exposed to moist air to ensure formation of a surface oxide film. Where no oxide film was present, corrosion of the steels by the fuming acid was increased in the presence of the inhibitor: the corrosion became more extensive as the chromium content of the steel fell below 15 per cent.

Liquid-phase corrosion of carbon steel was inhibited by hydrofluoric acid if a surface film was present when the steel was exposed, but, since conditions necessary for passivation are highly critical, this material is not recommended for use with fuming nitric acid.

Corrosion of tantalum and of the titanium alloys tested was increased by the addition of hydrofluoric acid to liquid fuming nitric acid. The corrosion-resistance of chromium, A nickel and lead was, however, slightly improved by such addition and these materials are considered suitable for use with inhibited acid, though in the gas phase the corrosion of chromium was found to be slightly increased by the inhibitor.

Storage of fuming nitric acid, in the gas and liquid phases, for prolonged periods under fluctuating-temperature conditions, did not impair the inhibiting effect of hydrofluoric acid on the corrosion of the aluminium alloy and the niobium-stabilized stainless steel. These materials also displayed good resistance to corrosion by NO_2, in both the gas and liquid phases. Carbon steel was slightly attacked by that medium.

Although in the case of aluminium alloys the inhibiting effect of hydrofluoric acid on corrosion of fuming nitric acid appears to be due to formation of an adherent surface film of an aluminium-fluorine compound, the passivation mechanism operative with alloy steels has not yet been clearly established. Work is in progress on this aspect.

Stainless Steel in Sulphuric Acid: Effect of NO, HNO_2 and HNO_3

W. P. McKINNELL, L. F. LOCKWOOD, R. SPEISER, R. H. BECK and M. G. FONTANA: 'The Effect of NO, HNO_2 and HNO_3 on Corrosion of Stainless Steel by H_2SO_4.'

Corrosion, 1958, vol. 14, Jan., pp. 9t-12t.

Stainless steel of the 18-8 type may be passive or active in a 10 per cent. solution of sulphuric acid, depending on the history of the surface of the steel. Pickled surfaces subsequently exposed to air are passive, while abraded surfaces are active. The steel used in the tests described in this paper was a 0·1 per cent. carbon 18-8 type. Earlier work by other investigators had shown that nitrous acid is involved in the passivation of iron in concentrated nitric-acid solutions and the present work was designed to demonstrate possible passivation effects which might result from addition of NO, HNO_2 and HNO_3 to sulphuric-acid solutions.

The experiments carried out were of two types: (1) corrosion tests including time and potential measurements, and (2) sorption tests. Descriptions of the potential-measuring equipment, the sorption apparatus and the corrosion-testing set-up are illustrated.

It was found that specimens which had been activated by abrading and were then exposed to 10 per cent. H_2SO_4 solutions were passivated by bubbling nitric oxide through the solution. Passivation is apparently due to the presence of HNO_2 in the acid: bubbling of oxygen through the solution did not passivate the abraded specimens.

Abraded steel exposed to an atmosphere of nitric oxide before immersion in nitric-oxide-free H_2SO_4 remained active, showing that adsorption of nitric oxide is not alone responsible for the passivation. Removal of HNO_2 from the solutions by addition of urea destroys the passivating effect of the nitric-oxide treatment of H_2SO_4 solutions.

The addition of one part of 10 per cent. HNO_3 to 60 parts of 10 per cent. H_2SO_4 solution has a similar passivating effect and this passivation also is destroyed by addition of urea. The authors conclude that neither nitric oxide nor HNO_3 alone causes the passivation; the presence of HNO_2 is essential.

Corrosion of Stainless-Steel Tubing

J. DEDIEU and L. PENNEC: 'Analysis of Types of Corrosion Occurring in 18-8 Stainless-Steel Tubing. Causes and Preventive Methods.'

Corrosion et Anticorrosion, 1957, vol. 5, Nov., pp. 348-58.

The service life of components made from 18-8-type chromium-nickel stainless steels can be greatly reduced by misunderstanding of the types of application for which they are suitable, or by ignorance of the precautions which must be taken to derive full advantage from their use. In this paper specific examples of corrosion failures which have occurred in tubing fabricated from such steels is used as a

basis for discussion of some of the factors which experience has shown to be essential for optimum performance. Rust staining, pitting, knife-edge corrosion and stress-corrosion are thus discussed, and in each case the causes of the attack are analyzed and preventive measures are recommended.

Pickling of Stainless Steels

W. E. McFEE: 'Pickling Stainless to Remove Scale.' *Steel*, 1957, vol. 141, Nov. 25, pp. 103–4, 107.

The article discusses the mechanical and chemical techniques generally employed to remove scale which forms on stainless steels during welding or as a result of exposure to furnace gases and high temperatures during thermal treatment. A brief outline is given of the conditions in which mechanical methods (shot blasting, tumbling) may be used, and the major part of the article is devoted to consideration of pickling treatments. Details are given of the compositions of suitable solutions and operating procedure, and recommendations are made with respect to the precautions necessary to secure optimum results.

Lubrication of Stainless Steel by Liquid Sodium

R. B. CAMPBELL: 'Liquid Sodium as a Lubricant.' *Instn. Mechanical Engineers' Conference on Lubrication and Wear*, Oct., 1957, *Paper* 56; 5 pp.+figures.

In certain types of atomic power plant in which sodium is used as a heat-exchange medium it may be desirable that circulation should be by means of mechanical pumps completely enclosed within the system. The pump bearings would thus be required to operate with liquid sodium as a lubricant. Wear, whether initiated by mechanical or corrosive means, must be avoided, because (*a*) the pumps have to function without attention for a period of years, and (*b*) loose wear débris must be kept to a minimum, on account of possible complications which it would cause in other parts of the system. (Conventional bearing materials, which normally contain a high proportion of tin, antimony or lead, are unsuitable.)

In view of the proved suitability of austenitic chromium-nickel steel for many applications in atomic energy plant, the behaviour of this material when lubricated with sodium was investigated. The rubbing specimens comprised two stainless-steel rings and the tests were carried out in an oxygen-free nitrogen atmosphere. The sliding speed in all the tests was 20 ft./min., and, in order to lessen the possible effect of any corrosive action by the liquid sodium, the tests were of short duration (maximum running time about 40 min.) and were confined to the temperature range 100°–200°C.

Results of first experiments indicated that the surface condition of the stainless steel might influence lubrication, but later tests on specimens having various types of surface finish demonstrated no statistically significant difference in performance traceable to that variable.

The results suggest that stainless-steel surfaces lubricated with liquid sodium are capable, in the temperature range used, of supporting moderate loads without development of severe failure. Wear can occur to an appreciable extent, but due to the high thermal conductivity of the lubricant, local overheating, and consequent seizure, does not take place.

Some tests in which stainless steel was lubricated with a medium-grade mineral oil are compared with those in which sodium was the lubricant. It was found that, with respect to the influence of surface finish, sodium lubricates in a reverse sense to mineral oil: best lubrication with sodium is obtained on turned surfaces and less satisfactory results were observed with smoother abraded surfaces, whilst for mineral oil the order of effectiveness is reversed.

Nickel-Aluminium-Bronze Marine Propellers

See abstract on p. 76.

Austenitic Steel Electrodes for Metal-Arc Welding

BRIT. STANDARDS INSTN.: 'Chromium-Nickel Austenitic Steel Electrodes for Manual Metal-Arc Welding.' *B.S.* 2926: 1957; 16 pp. Price 4/6.

The specification is one of a series of British Standards for covered electrodes for metal-arc welding (see also *B.S.* 639, 2493 and 2549). It specifies weld-metal compositions controlled with reference to contents of carbon, silicon, manganese, nickel, chromium, molybdenum, niobium, tungsten, sulphur and phosphorus. The types included are 18-8 unstabilized, 18-8-Nb, 19-10-Mo, 18-10-Mo-Nb, 25-12, 25-12-W, and 25-20 chromium-nickel steels.

Classification symbols are allotted to the various types of electrode, to indicate chemical composition and their suitability for d.c. and/or a.c. welding. Requirements are laid down also for size of electrode, tolerances on diameter of core wire, characteristics of flux, code marking and inspection. Full details (illustrated by figures) are given of tests called for to ensure correct chemical composition of the weld metal and quality of welds.

Appendix A presents a table showing alternative sizes of electrodes not supplied to measurements commonly used by British manufacturers.

Appendix B makes recommendations for preparation of etched specimens.

Appendix C contains a drawing of the standard proportional round test pieces.

Constitution of Nickel-Boron Alloys for Use in Brazing

G. S. HOPPIN: 'A New Nickel-Boron Phase Diagram for Brazing-Alloy Developments.' *Welding Jnl.*, 1957, vol. 36, Dec., pp. 528s–30s.

In recent years there has been increasing development and use of oxidation- and corrosion-resisting nickel-base brazing alloys of the nickel-chromium-silicon-boron, nickel-silicon-boron, and nickel-chromium-silicon types. Most of the boron-bearing alloys were originally designed to be used as hard-surfacing materials, and among them the nickel-chromium-silicon alloy was specifically developed to eliminate boron, which cannot be tolerated

in certain nuclear-power plant, due to its high neutron-capture cross-section. For certain applications in jet engines, where brazing of very thin heat-resisting materials is involved, all these alloys possess certain drawbacks: the boron-containing alloys tend to dissolve the base alloys to an excessive extent and the boron-free alloy has an extremely high brazing temperature (2200°F.: 1205°C.).

On the presumption that boron-containing alloys could be extended in application if the boron content were lowered from the 2·5–4·5 per cent. level which had been selected for hard-facing materials, work was initiated by the General Electric Company to develop modified brazing alloys. Study of phase relationships at the nickel-rich end of the system indicated that some of the data in published literature are incorrect. Re-investigation was therefore undertaken, and this paper contains a modified phase diagram (boron 0–4 per cent.) resulting from these studies.

The major features of note in the revised diagram are (1) a eutectic temperature of 1980°F. (1082°C.), (2) a eutectic composition of about 3·5 per cent. boron (by extrapolation), and (3) a maximum solid solubility of boron in nickel of 0·7 per cent. at 1980°F. (1082°C.), declining to 0·3 per cent. at 2225° and 1925°F. (1218° and 1051°C.). The findings of the authors are compared with the form of the diagram given by Giebelhausen in 1914, which is believed to be the only one available in the literature.

It is pointed out that in connexion with brazing alloys the following newly established facts are significant. Firstly, the eutectic temperature, as determined by the authors, is 100°F. (55°C.) lower than has previously been reported, and secondly, it has been established that boron has a finite solubility in nickel. Knowledge of the true eutectic temperature is of importance in certain jet-engine and rocket applications, in which temperatures of the order of 2000°F. (1095°C.) may be reached on brazed parts. The eutectic temperature and composition will also serve as a guide in determining additions of boron which can be made to lower the melting point of nickel-base high-temperature brazing alloys. Further, in order to achieve maximum high-temperature strength in nickel-alloy-brazed joints, diffusion treatments to remove boron from the joint are sometimes employed. The establishment of the limits of the boron-solid solution in nickel will assist in indicating the boron levels permissible in nickel-alloy-brazed joints for avoidance of re-melting of the eutectic at 1980°F. (1082°C.).

Alloys for Brazing Inconel to Stainless Steel

G. H. SISTARE and A. S. McDONALD: 'Oxidation-Resistant Brazing Alloys.'

Welding Research Council Bull. No. 30, Sept. 1956; 13 pp.

The report gives the results of a comprehensive investigation carried out in the Handy and Harman Research Laboratory, on behalf of Wright Development Center. The purpose of the work was the development of brazing alloys suitable for use in joining heat-conducting metal fins to Inconel tubing intended for service at temperatures of the order of 1400°–1600°F. (760°–870°C.). The fin materials were stainless-steel-clad copper and oxidation-hardened silver-magnesium-nickel alloy (99·5-0·3-0·2 per cent.).

The report opens with a discussion of the requirements which must be met by a brazing alloy to be used for such purposes, and of the 'specification' which formed the essential basis of the research. A highly detailed account is then given of tests made on alloys which might be expected to match up to the requirements. The compositions studied were evaluated according to the following procedure: specimens of the alloys were exposed in air at temperatures in the range 1300°–1600°F. (705°–870°C.), for times up to seven days, to determine their oxidation-resistance. Alloys which proved satisfactory in these tests were subjected to pad tests (in which fin and tube materials were used as the pad) and were used to prepare butt and lap joints and, in some cases, fin and tube assemblies. These joints and assemblies were then subjected to high-temperature exposure and, on the basis of the results, short-time tensile tests, at high temperatures, were made on promising types.

The alloys examined were selected on the basis of industrial experience and of deductions from equilibrium diagrams. In each series factors affecting choice of composition are discussed. Full details are given of results obtained from investigation of the systems shown below: exact compositions of the alloys tested are given in all cases.

- Silver-manganese
- Silver-aluminium and silver-aluminium-zinc
- Silver-magnesium and silver-magnesium-zinc
- Silver-gold and silver-gold-aluminium
- Silver-palladium and silver-palladium-aluminium
- Silver-silicon
- Silver-copper-palladium, with or without one of the following elements: aluminium, chromium, silicon, magnesium and beryllium
- Gold-nickel and gold-nickel-chromium
- Copper-nickel-manganese, with or without chromium, aluminium, beryllium or silicon
- Copper-aluminium-tin
- Palladium-aluminium-gold
- Nickel-indium and nickel-chromium-indium
- Palladium-nickel containing varying amounts of chromium, silicon, or phosphorus, separately or in combination

The 'seeding' tests indicated that only the gold-base and palladium-nickel-base alloys were sufficiently promising for further study. Both the 82·5-17·5 gold-nickel and the 72-22-6 gold-nickel-chromium alloys form tough ductile solid solutions which could be worked to wire or sheet. Such alloys proved resistant to oxidation and are considered to be suitable for brazing Inconel to stainless steel without flux in a protective helium atmosphere.

Work on the palladium-nickel-base alloys centred on four systems within the composition limits shown above, and varying in Pd/(Pd+Ni) ratio.

Extensive tests made to determine the influence of that ratio on the melting range and working temperature led to the choice, for further work, of alloys containing 45 per cent. Pd/(Pd+Ni). It was found that oxidation-resistance improved with increasing silicon and chromium contents, but that it was impaired by increasing Pd/(Pd+Ni). The following compositions proved suitable for brazing Inconel to stainless-steel-clad copper, at brazing temperatures in the range 1900°–1950°F. (1040°–1065°C.):

Pd	Ni	Si	Cr
%	%	%	%
36·9	45·1	8	10
36·45	44·55	9	10

These compositions were chosen as a compromise, to give satisfactory working temperature and melting range, but later work led the authors to the conclusion that some alloys having a 30 per cent. Pd/(Pd+Ni) base, with additions of phosphorus and/or boron, may offer even better prospects of obtaining the desired properties.

Welding of Nickel-Alloy Heat Exchangers

P. PATRIARCA, G. M. SLAUGHTER and W. D. MANLY: 'Heat-Exchanger Fabrication.'

Welding Jnl., 1957, vol. 36, Dec., pp. 1172–8.

Fabrication of heat-exchanger units has assumed additional importance with the advent of nuclear-power plants. One of the most important joints in any heat exchanger is that used to effect a leak-tight seal between the tube and the tube sheet, and the nature of the service in most atomic energy plants makes the welded tube-to-tube sheet joint essential. The conditions existing in such plant have necessitated development of highly specialized methods of fabrication, to satisfy the stringent requirements specified, and to ensure trouble-free service without the possibility of normal maintenance.

This paper describes, with informative illustrations, design and welding + back-brazing procedure developed at the Oak Ridge National Laboratory. The method is demonstrated as applied to heat exchangers of Inconel (nickel-chromium-iron alloy).

It is stated that prototype components consisting of compact, relatively complex, assemblies of thin-walled, small-diameter tubing produced by the methods described have performed satisfactorily in extensive simulated-service tests. The authors recommend that the principle of welding and back-brazing should be incorporated into heat-exchanger fabrication in all cases where leak-tightness is essential throughout service life.

Welded Austenitic/Ferritic Steel Joints

J. E. DONAHUE: 'Butt Welding Austenitic Stainless Steel to Ferritic Steel in Cylindrical Shapes.'

Welding Jnl., 1957, vol. 36, Nov., pp. 1074–7.

Several methods have been proposed for the joining of austenitic to ferritic steel, as required, for example, in components operating in steam-power stations. Reference is given to reports in which the respective methods are described, and some experience of their usefulness is reported.

The present paper describes design and manufacturing techniques developed by Westinghouse Electric Corporation, Philadelphia, Pa., for production of transition sections suitable for making such joints. The sections are made by a carefully controlled butt weld between the two types of steel. Application of the design and method is illustrated by reference to typical joints in turbine plant.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks.*

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